REPORT DOCUMENTATION PAGE

Form Approved OMB No. 0704-0188

1. REPORT DATE *(DD-MM-YYYY)* 23-09-2002

2. REPORT TYPE Final Report

3. DATES COVERED *(From – To)* 11-Sep-01 - 07-Nov-02

4. TITLE AND SUBTITLE
Processing of submicron-grained billets of Ti-6Al-4V titanium alloy by multiple forging

5a. CONTRACT NUMBER ISTC Registration No: 2124p

5b. GRANT NUMBER

5c. PROGRAM ELEMENT NUMBER

5d. PROJECT NUMBER

5d. TASK NUMBER

5e. WORK UNIT NUMBER

6. AUTHOR(S)
Professor Gennady Salishchev

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)
Institute for Metals Superplasticity Problems
Khalturin str.39
Ufa 450001
Russia

8. PERFORMING ORGANIZATION REPORT NUMBER
N/A

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)
EOARD
PSC 802 BOX 14
FPO 09499-0014

10. SPONSOR/MONITOR'S ACRONYM(S)

11. SPONSOR/MONITOR'S REPORT NUMBER(S)
ISTC 00-7051

12. DISTRIBUTION/AVAILABILITY STATEMENT
Approved for public release; distribution is unlimited.

20030130 104

13. SUPPLEMENTARY NOTES

14. ABSTRACT

This report results from a contract tasking the Institute for Metals Superplasticity Problems as follows: The project will study sub-microcrystalline structure formation in a Ti-6Al-4V titanium alloy during large plastic deformation by multiple isothermal forging; and to produce large (diameter up to 200mm) billets with homogeneous microstructure and a grain size of no more than 0.5 microns.

The results of the project will establish the connection between microstructural changes resulting in the formation of a sub-microcrystalline structure and resulting mechanical behavior in a Ti-6Al-4V alloy during large plastic deformation at temperatures about half the melting temperature and reveal whether dynamic recrystallization is the pertinent metallurgical process. The influence of temperature and rate of deformation as well as the initial microstructure (grain size, phase volume fraction, thickness of plates of α and β-phases) on the kinetics of structure formation, its uniformity, and mechanical properties will be shown. The influence of initial microstructure on cavitation and workability will also be determined.

Based on the experimental studies, computer modeling of multi-step isothermal forging in a special die set unit will also be conducted. Optimum routes of multiple isothermal forging in the special die set will be determined.

15. SUBJECT TERMS
EOARD, Materials, Metallurgy & Metallography

16. SECURITY CLASSIFICATION OF: a. REPORT	b. ABSTRACT	c. THIS PAGE	17. LIMITATION OF ABSTRACT	18, NUMBER OF PAGES	19a. NAME OF RESPONSIBLE PERSON
UNCLAS	UNCLAS	UNCLAS	UL	53	Charles H. Ward, Lt Col, USAF

19b. TELEPHONE NUMBER *(Include area code)* +44 (0)20 7514 3154

Final

Project Technical Report

of ISTC 2124p

" Processing of submicron-grained billets of Ti-6Al-4V titanium alloy by multiple forging "

(From September 1, 2001 to August 31, 2002 for 12 months)

Salishchev Gennady Alekseevich

(Project Manager)
Institute for Metals Superplasticity Problems

September 2002

This work was supported financially by European Office of Aerospace Research and Development, UK, and performed under the contract between the International Science and Technology Center (ISTC), Moscow, European Office of Aerospace Research and Development and the Institute for Metals Superplasticity Problems, Russia.

CONTENT

1. Project Information

"Processing of submicron-grained billets of Ti-6Al-4V titanium alloy by multiple forging"

(From September 1, 2001 to August 31, 2002 for 12 months)

Salishchev Gennady Alekseevich (Project Manager)

Institute for Metals Superplasticity Problems

Abstract

The method of an isothermal multiple step forging for producing large scale billets out of Ti-6Al-4V alloy with sub microcrystalline grain size less than 0.5 μm has been developed. The influence of deformation temperature on formation of globularized microstructure was studied in the titanium alloy Ti-6Al-4V with initial coarse-grained martensite type structure. It has been shown that the decrease in deformation temperature decreases the size of forming globularized grains and increases critical strains of their initiation. The grain size less than 0.5 μm is formed at the deformation temperature not exceeding 600°C and the strain rate $10^{-3}s^{-1}$.

Mechanical behavior and evolution of microstructure of the alloy Ti-6Al-4V with initial coarse-grained martensite type microstructure were studied in the process of successive compressive straining of samples in three orthogonal directions at 800 and 550°C and the strain rate $10^{-3}s^{-1}$. It has been shown that true flow stress-total deformation (S-Σe) curves for both temperatures are similar. They have a peak of flow stress, and stages of softening and steady flow. The value of the strain rate sensitivity coefficient m and the value of activation energy in the stage of steady flow are close to the superplastic deformation. These testify the development of this process in the final stage of transformation of the coarse-grained lamellar microstructure to the globular one. The results obtained demonstrate that it is possible to use successive deformation in three orthogonal directions for formation of homogeneous SMC structure in billets.

It has been studied the effect of the initial microstructure type of the alloy Ti-6Al-4V. It has been established that for formation of homogeneous SMC structure it is more preferable to use the alloy in the condition with the initial martensite structure or perform intermediate processing of the alloy for formation of globular type microstructure where areas of β-transformed constituents are absent.

The effect of the initial microstructure of the alloy Ti-6Al-4V on its workability was studied. It has been shown that the alloy with initial martensite type structure displays higher plasticity and larger strains until formation of cracks than the alloy with globular-lamellar structure. The alloy with a globular type microstructure demonstrates the highest workability.

Performance of mathematical 3D modeling using DEFORM-3D for determination of the optimal route of the technological process and the geometry of the special die tooling to produce massive billets out of the alloy Ti-6Al-4V with homogeneous microstructure and a grain size less than 0.5 μm was done. The results obtained provide determining the most optimal die set geometry, the installation of the billet in the die set and the sequence of strain operations

Using the method developed two large size billets, ∅150×200 mm, with homogeneous microstructure were produced. Analysis of their quality has shown that the homogeneous microstructure with a grain size less than 0.5 μm was formed in them. Investigations of mechanical properties have shown that strength and ductile characteristics of the billets produced are close in radial and tangential directions.

Project activities are described in three papers, and presented at 2 Conferences (three presentations). RU Patent application was filed.

Keywords: Severe Plastic deformation, Titanium alloy, Superplasticity, Dynamic globularization, Lamellar structure, Globular structure, Sub microcrystalline structure, Modeling, Method, Multiple step isothermal forging, Project, Die set.

The work has been performed by the following institutes and partners:

Participated institute:
Institute for Metals Superplasticity Problems of Russian Academy of Sciences
39, ul. Stepana Khalturina, Ufa 450001, Republic of Bashkortostan, Russia
Tel: (3472)-253712
Fax: (3472)-253-759
E-mail: gensal@imsp.da.ru

Foreign Partner:
European Office of Aerospace Research and Development,
223-231 Old Marylebone Road, NW1 5TH, London, UK
Project leader: Dr. Charles H. Ward, Lt Col, USAF
Program Manager for Structural Materials
European Office of Aerospace Research & Development
Tel: +44 (0)207 514 3154 DSN (314) 235-3154
Fax: +44 (0)207 514 4960
E-mail: charles.ward@london.af.mil

Duration of the Project: 12 month.

Total Cost of The Project: $75, 000

2. Introduction

2.1. Current status of the problem.

One of the advance directions of materials science is the development of materials with a sub microcrystalline (SMC) structure. These materials have a grain size of less than 1 μm and show enhanced mechanical properties such as increased strength and fatigue resistance. They also show superplastic behavior at temperatures much below the temperature range typical for materials with micron-sized grains; this can lead to a decrease in processing tool costs and reduction of energy and material consumption [1]. Hence, processing of SMC materials and investigation of their properties are very urgent problems. One of the most promising methods for producing large-scale billets with SMC structure is severe plastic deformation (SPD) up to true strain values no less than 6-8. The nature of SMC structure formation resulting from severe plastic deformation is not clear yet. A number of authors [2,3] have postulated that the formation of SMC grains during plastic deformation is due to a process similar to discontinuous dynamic recrystallization (DRX); where there occur bonding and serration of separate parts of grain boundaries and formation of new grains at initial grain boundaries. Others [4,5] explain SMC structure formation as a result of the continuous DRX due to formation of substructure, interaction of subboundaries with dislocations and formation of high angle grain boundaries. It is known the third point of view. The authors [6-8] show that during cold deformation new grains are resulted from formation and intersection of deformation origin boundaries with high angle misorientation.

In the case of the first two concepts on the mechanism of grain formation it could be used the following dependence of the size (d) of recrystallized grains on the steady flow stress σ_s for analysis of conditions of DRX occurrence:

$$\sigma_s=kd^{-n}, \qquad \text{(Eq. 1)},$$

where k is an empirical constant, and the grain size exponent $n=0.5\div1$ [9]. It is assumed that $n\approx0.5$ is achieved when high angle grain boundaries are formed, while $n\approx1$ when subgrains are formed. It is seen that the size of dynamically recrystallized grains can be decreased by decreasing the temperature or increasing the strain rate because in both cases that leads to an increase in the flow stress. The decrease in the temperature is more preferable since in this case it easier to control grains growth after completion of deformation. Grains produced during DRX are almost the same size as subgrains, while the subgrains themselves can be as small as several tens of nanometers [10]. Consequently, SPD can results in formation of the grain size close to nanocrystalline. The authors [5,7] have shown that for titanium over the temperature range of cold deformation n is equal to 0.33 and at high temperatures n=1.1. The results for ferritic and austenic steels and copper are similar [11]. There occurs an apparent change in the mechanism of new grain formation. The dependence mentioned above has no such a physical meaning for two-phase alloys as for one-phase ones. Processes of dynamic recrystallization and dynamic globularization are distinguished significantly. Under the DRX occurrence there is a replacement of stages of strengthening (increase in dislocation density) and softening (recrystallization) in metals [9]. According to literature data [12,13] in case of dynamic globularization at high temperatures there takes place continuous softening (rotation of plates in the direction of plastic flow, separation of plates and spheriodization of parts of the plates) that results in a steady stage flow where features of superplastic deformation are observed. That is why the value n in this case has no the same physical meaning as for one-phase materials. Nevertheless, one can assume that analysis of this relationship at different deformation temperatures would allow to establish whether the mechanism of globularized grain formation change in the range of SPD temperatures. Note that similar to titanium the decrease in the deformation temperature in two-phase titanium alloys leads to significant structure refinement. In particular, a SMC structure with d of about 0.06 μm has been formed in Ti-6.0Al-3.2Mo-0.4Fe(wt.%) ($\alpha+\beta$)-titanium alloy at the deformation temperature T=550°C [14] and in hydrogen doped two-phase Ti-6.0Al-3.2Mo-0.4Fe alloy after deformation at T=525°C the grain size d=0.04 μm [15].

In general the loss of lamellar structure stability at high temperatures exposure associated with the unbalanced state of surface tension force in the subboundary/boundary and interphase surface intersection [16-18]. Surface tension force violate the equilibrium in the vicinity of 180 dihedral angle formed by these boundaries. This results in local dissolution of α-phase in the α/α boundaries and the α/β interphase boundary intersection. Note that in titanium alloys the lamellar structure is rather steady to thermal exposure. The paper [19] assumes that this fact is the result of high level of interphase energy anisotropy of this system. The history of material processing also influences significantly on stability of plates to spheriodization. The authors [20-22] have shown dependence of globularization from a sort of microstructure. The kinetics of globularization during hot deformation accelerates essentially as the thickness of plates and the size of β-grains decrease [21]. Without doubt, the bending of interphase surface via its division by shear bands will increase its energy and contribute to dissolution of plates [23,24]. The presence of twins in α-phase [22] and formation of transverse high angle boundaries and subboundaries [23] also contribute to that. The rate of dissolution of α-plates is generally determined by the balance between the surface energy of α/α boundary and that of the interphase one [25,26]. So, the formation of transverse high angle boundaries is effective as well as the transformation of conventionally semi-coherent interphase boundaries into non-coherent ones due to their interaction with dislocations [19]. In the process of loading the plates turn in the direction of deformation. This fact was identified in the paper [27] where a model based on diffusion creep in one or both phases was proposed. This diffusion creep explains the main stage of plate globularization, namely their division. A number of papers [24,28,29] also noted that at some definite stage of transformation of the lamellar microstructure to the equilibrium one the strain rate sensitivity coefficient (m) sharply rises and achieves values corresponding to superplasticity. This testifies that at this stage of deformation grain boundary sliding makes its contribution to microstructure transformation. As a result, there occurs a turn and shift of both plates and their fragments. Local superstresses occurring leads to the increase in the density of defects that contribute to acceleration of diffusion and dissolution of plates. And finally spheriodization also can contribute to polymorphic α→β transformation which can be resulted from both the local superstresses [30] and the initial non-equilibrium of the phase composition of the deforming alloy [31].

Note that dynamic globularization occurs more fully only within the definite temperature-strain rate range close to superplasticity. As the strain rate increases the plastic flow is localized in coarse shear bands [24] that leads to formation of a rather heterogeneous structure and requires the increase in the strain value and introduction of intermediate annealing operation. With decreasing the strain rate the role of diffusion process becomes more important. As a result, α-phase plates become more coarse that retards grain boundary slip and transformation of grains to globular ones.

It should be noted that critical deformation required for initiation of both DG and DRX increases significantly with decreasing temperature. So, large strain values are necessary to form completely globularized SMC structure. Moreover high stresses and low ductility are observed within the temperature range of deformation. This, in turn, retards processing and formation of completely globularized microstructure. That is why, it is necessary to search methods and regimes of deformation which can provide most efficient processing.

It is known several methods for processing SMC structure in massive billets via SPD. One of them is the method of equal channel angular extrusion (ECAE) [1]. The ECAE method is used most often for ductile metals at room temperature. In recent years, results on titanium and its alloys deformed by ECAE at elevated temperatures have been obtained [1, 32-34]. The increase in the temperature for processing titanium and its alloys by ECAE is necessary to decrease loads and increase workability. However, there are no data on possibility of producing large scale semi manufactured products by means of this method.

Multiple isothermal forging has also been developed for producing SMC structures in titanium base alloys at high temperatures in the (α+β)-region [20]. For obtaining SMC structure, it is necessary to decrease the temperature of deformation; however, ductility is decreased in these instances as well. This is why the decrease in the temperature of deformation can be performed

step-by-step that results in formation of a recrystallized/globularized microstructure at each step. The microstructure formed, in turn, can lead to the increase in plasticity at a lower temperature of the following stage. Strain localization at low forging temperatures is thus avoided as a fully recrystallized microstructure is obtained.

Due to the non-uniform nature of plastic flow DRX/DG occurs, first of all, in areas subjected to the most intense plastic deformation. Deformation uniformity is attained by increasing the number of forging steps at each deformation temperature and/or by using a special die geometry which increases the uniformity of plastic flow. At the same time, it is very important to take into account the influence of the initial structure of the material on its workability. The problem of alloy workability is especially important because of the need to utilize low temperatures to obtain the desired SMC structure. From literature data it is known [35,36] that preliminary quenching of billets from β-region is most preferable for formation of the most fine-grained and homogeneous microstructure. The question of workability and uniformity of plastic flow is most important in case of large billets. Investigations of this kind are almost nonexistent.

Thus, research is needed to relate the formation of SMC structure in titanium alloys such as Ti-6Al-4V to the conditions of severe plastic deformation by multiple isothermal forging; to reveal the role of structural and technological factors in formation of uniform microstructure; and to conduct modeling of the forging process to produce large (∅150×200mm) billets with homogeneous microstructure and a grain size of no more than 0.5 μm.

2.2. Objectives of the Project.

1. Determination of structure evolution the Ti-6Al-4V alloy with initial lamellar microstructure and its mechanical behavior during large plastic deformation within the temperature range 400-800 C and their conformity to the dynamic recrystallization/globularization process.
2. Determination of the influence of temperature –strain rate regimes of deformation, as well as the initial microstructure (e.g., grain size, phase composition, β- phase plate thickness), on the kinetics of SMC structure formation, its uniformity, pore formation and mechanical properties.
3. Computer modeling of multiple step forging for designing and manufacturing production tooling providing processing billets, Ø150x200 mm, with homogeneous microstructure and a grain size less than 0.5 μm.
4. Development of a pilot method for processing massive, ∅150×200mm billets with a grain size less than 0.5 microns. Production of a billet, ∅150×200mm, with a grain size less than 0.5 μm and evaluation of its quality.

Participation of specialists from Institutions involved in Project activities realization comprises the following:

Specialists of the Institute for Metals Superplasticity Problems, RAS have fulfilled the following tasks:

1. Determination of structure evolution the Ti-6Al-4V alloy with initial lamellar microstructure and its mechanical behavior during large plastic deformation within the temperature range 400-800 C and their conformity to the dynamic recrystallization/globularization process.
2. Determination of the influence of temperature –strain rate regimes of deformation, as well as the initial microstructure (e.g., grain size, phase composition, βphase plate thickness), on the kinetics of SMC structure formation, its uniformity, pore formation and mechanical properties.
3. Development of a 3D model for processing billet to determine the optimal geometry of die tooling at specified values of temperature, strain rate and strain value.
4. Production of a billet, ∅150×200mm, with a grain size less than 0.5 μm and evaluation of its quality.

Partner's representatives – Air Force Research Laboratory, AFRL/MLLM, Wright-Patterson Air Force Base, OH, 45433 (Dr. Lee Semiatin group) participate in the following:

1. Evaluation of structure and properties of billets with homogeneous fine-grained structure produced by the modernized method.

2. Search of ways for commercial implementation of the technology developed.

The cooperation resulted in development of the method for producing massive billets with ultrafine-grained homogeneous structure out of hard-to-deform titanium alloy Ti-6Al-4V and equipment for their production. The obtained results can be used in peaceful purposes.

2.3. Expected Results.

The investigation proposed deals with developing methods for processing materials and alloys with improved characteristics.

Realization of the Project activities planned will allow revealing microstructure evolution and mechanical behavior in alloy Ti-6Al-4V during large plastic deformation at temperatures about $0,4T_{ml}$ and evaluating their conformity to the dynamic recrystallization/globularization process.

The Project will allow establishing the influence of temperature –strain rate regimes of deformation, as well as the initial microstructure (e.g., grain size, phase composition, β phase plate thickness), on the kinetics of SMC structure formation, its uniformity, pore formation and mechanical properties..

Using the obtained results of microstructure investigations it is planned to development of processing regimes (temperature, rate, strain value) and routes for manufacturing bulk billets with an SMC structure by multiple isothermal forging

Taking into account the results of computer modeling of the distribution of stresses and strains within the billet interior during multiple isothermal forging it is planned to design and manufacture forging dies for processing a billet, ∅150×200mm, with homogeneous structure and a grain size no more than 0.5 μm by the method for multiple isothermal forging.

The specially developed production tooling and processing route will provide processing a billets, ∅150×200 mm, with homogeneous structure and a grain size no more than 0.5 μm.

2.4. Technical Approach and Methodology

Task 1: As the temperature of deformation decreases, the onset of recrystallization shifts to strain values $\varepsilon > 2$. In the case of usual types of uniaxial deformation (e.g., compression), such strain values can be attained only in zones of intense plastic flow of a sample. By contrast, large strain values of $\varepsilon = 6\text{-}8$ can be attained under multi-axis ('a-b-c') deformation conditions. A similar technique was used in the present study. Samples, ∅10×15 mm, with an initial lamellar coarse-grained microstructure were subjected to compression at continuously decreasing temperatures (800→400°C), $d\varepsilon/dt = 10^{-4}\ s^{-1}$, and reduction levels of approximately 80 pct. Some of the samples were subjected to a-b-c deformation. Before each rotation a regular shape was impared to the sample to determine flow stress at large strains. Concurrently, the evolution of microstructure and mechanical behavior were studied. Analysis of comformity of structural changes to the process of DR/DG was made by plotting the dependence of the steady state flow stress (σ_s) on the recrystallized grain size (d), according to the relationship $\sigma_s = kd^{-n}$. The temperature dependence of the critical strain for the initiation of recrystallized/globularized grains was analyzed too. The critical strain values for initiation of recrystallized/globularized grains were determined by the microstructure of the samples compressed by different degrees. Optical and electron microcopies were used for these investigations.

Task 2: The study of the influence of the initial microstructure on flow stress and recrystallized/globularized grain size was similar to the one described above. In addition, the volume fraction of recrystallized microstructure over the sample cross section was studied. To determine the workability and the influence of initial microstructure, the dependence of reduction in area and total elongation on deformation temperature was plotted. The total elongation results were necessary to determine allowable strains for free surface cracking during forging. Moreover, the influence of temperature of deformation and initial microstructure on the early stages of cavitation was determined. The data so obtained were used for determining the deformability of the alloy and

recommending optimal regimes of SMC structure formation. Tensile tests were performed on samples with a gauge diameter of 5 mm over the temperature range 550-800^{0}C.

Task 3: 3-D mathematical modeling was performed by a numerical method using DEFORM 3D. Properties of the material were described by Anand relationship. The constituent equation selected provided an opportunity to describe deformation behavior of a material being sensitive both to the strain value and the strain rate. As a result, diagram's for distribution of stress and strain intensities within the billet interior were plotted for each pass.

Task 4: For processing billets, ∅150×200 mm, with a grain size of no more than 0.5 μm and a homogeneous microstructure, firstly, a technological route based on multiple isothermal forging was identified. Such a procedure included the determination of the temperature of polymorphic transformation and the type of microstructure in various areas of a billet that was required for determing regimes of forging, i.e. temperature, strain rate, tolerable strain values, number of passes. After production of billets, the evaluation of microstructure quality was made using standard techniques of investigation.

3. Method, Experiments, Theory etc.

3.1. Materials for investigation.

As the research material a forged bar of diameter 230 mm made of titanium alloy Ti-6A1-4V was used. The bars were provided by the Verkhne-Saldinski Metallurgical Industrial Association. The chemical composition of the alloy under research is shown in Table 3.1.1. The polymorphic transformation temperature of the Ti-6A1-4V alloy bar 230 mm in diameter was 995 °C. The alloy macrostructure as received is shown on Fig. 1a. It indicates the coarse-grained composition of the bar as well as the fairly uniform grain distribution along its section. The average β-grain size equals 1000 μm.

Table 3.1.1.

Chemical composition of the Ti-6Al-4V alloy (wt.%, Ti-balance, residuals, max %)

Al	V	Fe	Si	Zr	C	O	N	H	others
6.3	4.1	.18	.029	.02	.010	.182	.010	.002	.08

The compression testing of the Ti-6Al-4V alloy was conducted in the "Schenck" RMS-100 testing machine at air atmosphere. The sample furnace exposure time before deformation amounted to 20 minutes in all the cases. Taking into consideration the samples deformation localization, all the structural research was conducted in their central areas. The tensile testing was conducted in the "Instron" universal dynamometer at air atmosphere. The heat time before deformation amounted to 15 minutes. In accordance with the testing results the yield stress ($\sigma_{0,2}$), ultimate strength (σ_B), reduction area (ψ), and aspect ratio (δ) were estimated.

The compression testing of Ti-6Al-4V alloy cylindrical samples for determination extreme deformation was conducted in the hydraulic press EU-100 with the maximum force of 1 MH. The press was equipped with isothermal die unit.

The large-scale billet with SMC structures were fabricated by the multiple step isothermal forging on a hydraulic press with the maximum force of 1600 t. The press was equipped with UIDIN-500 isothermal stamp block. The deformation strain rate amounted to ~$10^{-3}s^{-1}$.

3.2. Structural Research Methodology.

The microstructure research was conducted on the "Metaval", "Neophot-2", and «Axiovert 100" optical microscopes as well as on the "Epiquant" automatic structure analyzer. The fine microstructure was researched with the help of the JEM-2000 EX transmission electron microscope at the accelerating voltage 200 kV as well as with the JEM840 scanning electron microscope.

3.5. Mathematical Modeling Methodology.

3-D mathematical modeling was performed by a numerical method using DEFORM 3D. Properties of the material were described by Anand relationship. The constutuent equation selected provided an opportunity to describe deformation behavior of a material being sensitive both to the strain value and the strain rate.

4. The Results.

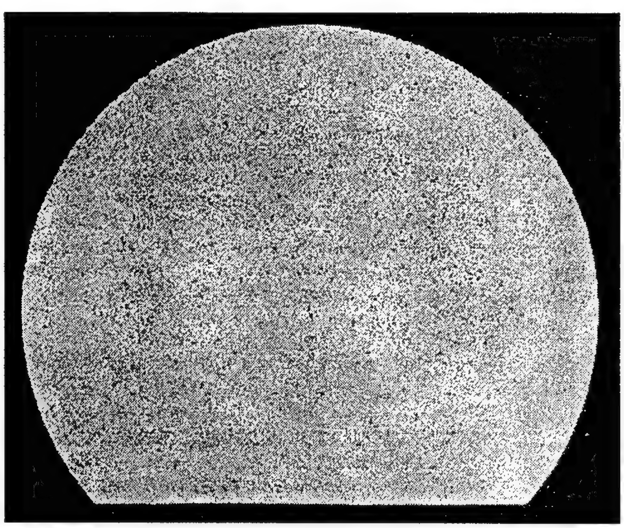
The project implementation took 12 months within which the efforts of the specialists previously engaged in mass destruction weapons development gave 620 man-days; the efforts of the material science specialists, as well as those of the support personnel gave 841 man-days. The above efforts totaled to 1461 man-days. The overall labor-output ratio of the project amounted to 73.05 man-months.

4.1. Technical achievements.

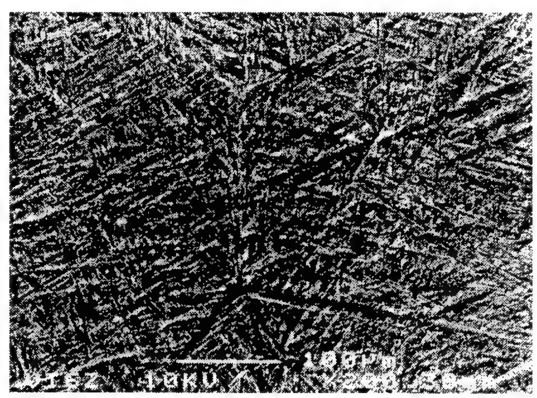
A-1 Task. Investigating the structural evolution of the Ti-6Al-4V alloy with initial lamellar structure and its mechanical characteristics under severe plastic deformations within the temperature range of 400-800°C, as well as their correspondence to the dynamic recrystallization/ globularization processes.

A-1.1. Investigating the temperature dependences of the flow stress on the size of the recrystallized/ globularized grains at the steady flow stage. Investigating the temperature dependence of the critical deformation for appearing of the first recrystallized/ globularized grains.

To conduct the research, the initial alloy bar was cut into billets 120 mm in length, which were then exposed to the multiple deformation at the β-area temperature of 1050÷1000°C to reduce the β-grains size. A microstructure with the average β-grain size of 250 μm was obtained. Next, the samples of 10 mm in diameter and 15 mm in height were cut out of the billets. Further, they were quenched in water from the β-area of T=1010°C, as a result of which α′-martensite structure was formed (Fig. 1b).



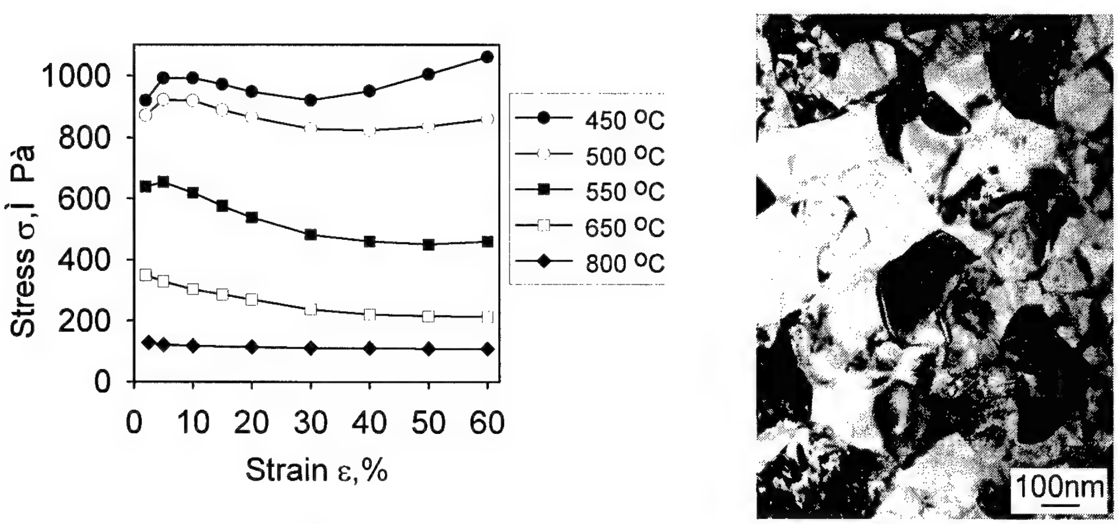
a



b

Fig. 1. Macrostructure of the alloy bar as received (a), microstructure of the Ti-6Al-4V alloy after quenching from the β-field (b).

After quenching, the samples were exposed to the compression within the temperature range of 400-800°C to study the temperature dependences of the flow stress on the size of the recrystallized/globularized grains at the steady flow stage, as well as the temperature dependence of the critical deformation for appearing of the first recrystallized/globularized grains. The σ–ε dependencies were plotted for the temperatures of 450, 500, 550, 650, 800°C, up to 70% deformation and $10^{-3}s^{-1}$ strain rate (Fig. 2a). The σ-ε curves for all the cases have the flow stress peak with the following softening and changing towards the steady flow stage. With decreasing of the deformation temperature, the flow stress of the alloy increases. At the deformation temperature of 450°C the shape of a σ-ε curve changes: the steady stage decreases and the repeated strengthening takes place. After the 70% deformation within the above-mentioned temperature range, there is a distinctly visible globularized structure formed in the alloy. The structure consists of the α- and β-phase grains. At 650°C and below the SMC structure is formed. It is characterized by the presence of a significant number of the fringe diffraction contrast, which is indicative of the lattice elastic distortion, as well as of the increased dislocation density in most of the grains (Fig. 2b). No signs of appearing of the recrystallized grains in phases were revealed. It is worth mentioning though, that with the prevalent α-phase amount at low temperatures and with the inevitable heterogeneity of the α-plates thickness, the recrystallization might take place in some of the thick plates. At 400°C the samples were ruptured at the early deformation stages due to their low plasticity. The relationship between the deformation temperature and the size of the globularized grains is shown on Figure 3. The globularized grains size decreases with decreasing of the deformation temperature, and at 450°C there is a microstructure formed with the grain size of approximately 0.1 μm.



a b

Fig. 2. Ti-6Al-4V alloy flow stress dependence on the strain at various temperatures and the strain rate of $10^{-3}s^{-1}$ (a); Ti-6Al-4V alloy microstructure after 70% deformation at 550°C and $10^{-3}s^{-1}$ (b). The initial condition is quenching from the β-area.

Using the data from Fig. 2a and Fig.3, the dependence of the flow stress σ at the steady flow stage on the globularized grains size d was determined. The dependence can be described as $\sigma=kd^{-n}$, where k and n are empirical constants (Fig. 4). For the alloy this dependence is fairly well approximated by the straight line with the exponent *n* approaching 1. However, the dynamic recrystallization (DR) and dynamic globularization (DG) processes differ fundamentally from each other. With the DR development in metal there is an interchange between the strengthening phase (increase in dislocation density) and softening phase (recrystallization) [5]. In case of DG at high temperatures, as stated in papers [7], continuous softening takes place (plates turning in the

direction of metal flow, their division and spheroidization) followed by the steady flow stage, within which the signs of superplastic deformation were noted. The *n* value in this case, therefore, does not have the same physical meaning as it has for single-phase materials. However, due to the constant value of the exponent *n*, it can be supposed that the mechanism of transforming a lamellar structure into a globular one in a two-phase Ti-6Al-4V alloy does not depend on the deformation temperature within the researched temperature range.

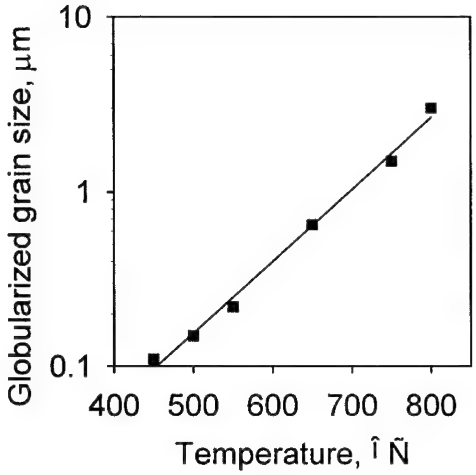


Fig. 3. Globularized grains size dependence on testing temperature in the Ti-6Al-4V alloy after 70% deformation at the strain rate of $10^{-3}s^{-1}$.

To determine the critical deformation necessary for forming of the new grains, the Ti-6Al-4V alloy cylindrical samples of ∅10×15 mm, quenched from the β-area, were exposed to compression. Issuing from the results of the earlier research, as well as from the published data [14], for each temperature of 550, 650 and 800°C a probable interval of the strain resulting in first grains forming was chosen. The samples were then compressed for different strains within the chosen intervals in every 10%. Next, the objects to be researched with the help of the electron microscope were cut out of the central areas of the compressed samples.

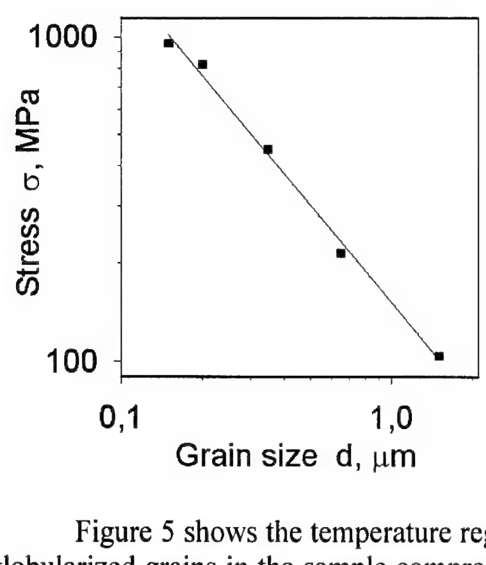


Fig. 4. Dependence of the flow stress σ at the steady flow stage on the globularized grains size d. The initial condition is quenching from the β-area.

Figure 5 shows the temperature regularity of the critical deformation for forming of the new globularized grains in the sample compressed at the deformation strain rate of 10^{-3} s^{-1}. It is noticed that decreasing the deformation temperature down to 550°C results in considerable increase of the strain necessary for forming of the new grains. Consequently, at lower temperature it is necessary to reach greater strain to form homogeneous submicrocrystalline structure.

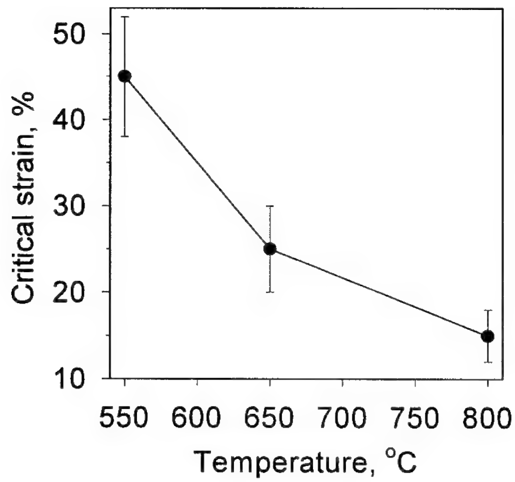
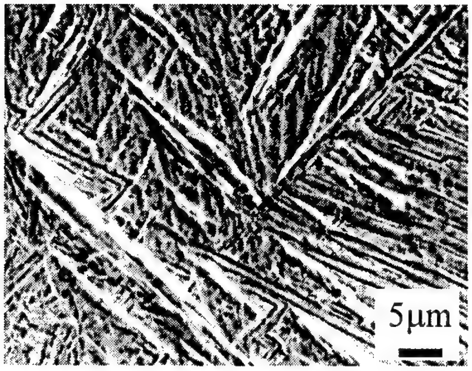


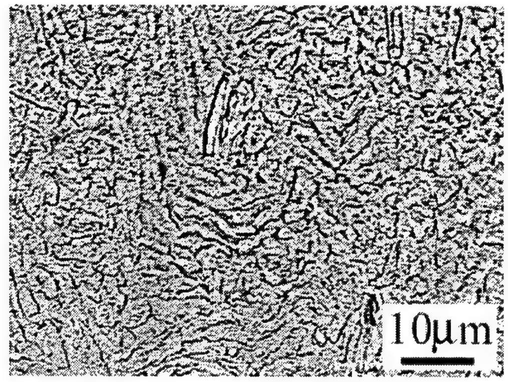
Fig. 5. Temperature regularity of the critical deformation for forming of the new globularized grains in the sample compressed at the deformation strain rate of 10^{-3} s^{-1}. The initial condition is quenching from the β-area.

A-1.2. Comparative investigation of the globularization kinetics at low and high deformation temperatures.

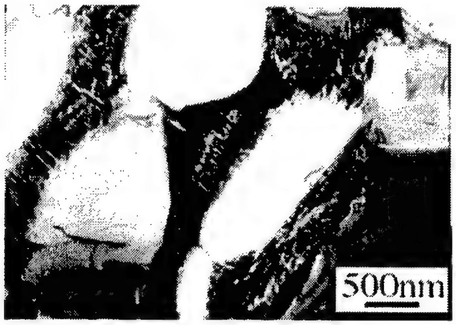
To research the Ti-6Al-4V alloy structure evolution in the process of deformation the same billets with the initial condition of quenching from the β-area were used. The samples of ∅10×15 mm were compressed by 15, 30, 50 and 70% with the initial deformation strain rate of 10^{-3} s^{-1} at the (α+β)-area temperatures of 550 и 800°C. The electron microscope research was conducted on the objects cut out of the central areas of the compressed samples. When heated to the deformation temperature, α′- martensite transforms into α- and β-phase plates, whose thickness decreases with decreasing of the heating temperature. Before deformation at 800°C the α-plate thickness equaled 1μm and the length was 10-40 μm (Fig. 6a). The α- and β-phase ratio was 70% by 30% correspondingly. The microstructure change along with increasing of the strain is similar to already described above [7, 18, 19, and 28]. Apparently, with its increase dislocation accumulation takes place in phases. It should be noted that tracing structural changes in β-phase because of the phase transformation on cooling from this deformation temperature was not successful. The shear bands, which bend the plates at the maximum deformation areas, were noticed in the plates colonies unfavorably oriented for deformation (Fig. 6b). Globular grains appear already at the degree of transformation of 20%. The further 30% deformation increase is accompanied by the recovery processes in phases. In the α-phase the dislocations are rearranged to form transversal sub-boundaries. Further, their orientation grows and they become high angular. Apparently, similar processes take place in the β-phase as well. Up to 50% deformation growth leads to turning of the α-plates in the direction of metal flow. Spheroidization of the α-plates divided into fragments, as well as fragments shifting with regard to each other takes place (Fig. 6d). The above is the evidence of grain-boundary sliding effect. At 70% the microstructure globularization process in the central area is for the most part completed. The α- phase globules are practically free of dislocations. The average grain size equals 2 μm (Fig. 6e).



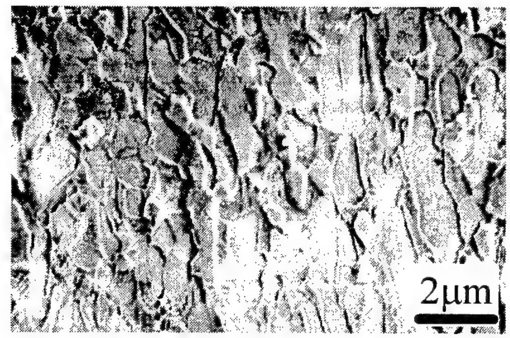
a



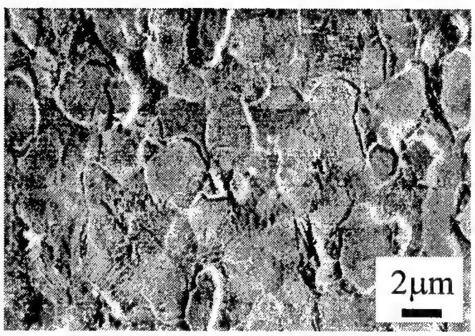
b



c



d



e

Fig. 6. The Ti-6Al-4V alloy microstructure before deformation (quenching from the β-area + 15 minutes aging at 800°C) (a). The Ti-6Al-4V alloy microstructure compressed at 800°C and $10^{-3}s^{-1}$ by 30% (b,c), 50% (d) и 70% (e).

Decreasing the deformation temperature up to 550°C does not lead to qualitative change of the processes under research. By the deformation start T=550°C and the aging of 25 minutes, the alloy has the lamellar structure, consisting of the coarse plates with thickness of approximately 0.3 μm and length of approximately 5μm, as well as of the smaller ones of the size of about 0.1×0.5 μm (Fig. 7a). The α- and β-phase ratio was 85% by 15% correspondingly. Within the colonies the coarse plates are placed nearly parallel to each other, with the small ones occupying the space between them. After deformation by ε=10% at 550°C and $10^{-3}s^{-1}$ the initial lamellar structure with distinct, slightly curved boundaries is generally preserved. Along the length of some plates a contrast change is revealed (Fig. 7b). On increasing deformation by 30%, dislocation accumulation and transversal low angular boundaries formation take place in the α-plates. Along the length of the β-phase interlayer the contrast change is also revealed, and their boundaries are curved in some parts. Turning of the most of the plates in the deformation direction occurs. Within the unfavorably oriented colonies an intensive shear deformation is noted, which results in plates bending (Fig. 7c).
The deformation up to 50% leads to forming a microstructure, in which separate grains of 0.2 μm with low dislocation density are encountered, as well as the plate residues, divided by the transversal boundaries into separate fragments (Fig. 7d). Grooves are noted at the cross of the interphase and intergranular boundaries. The amount of globular grains increases dramatically. Extended areas of various contrast sized from 0.2 to 0.8 μm with no distinct boundaries are also

noted in the microstructure. In some cases the pronounced plate fragments shifting with regard to each other was revealed (Fig. 7e).

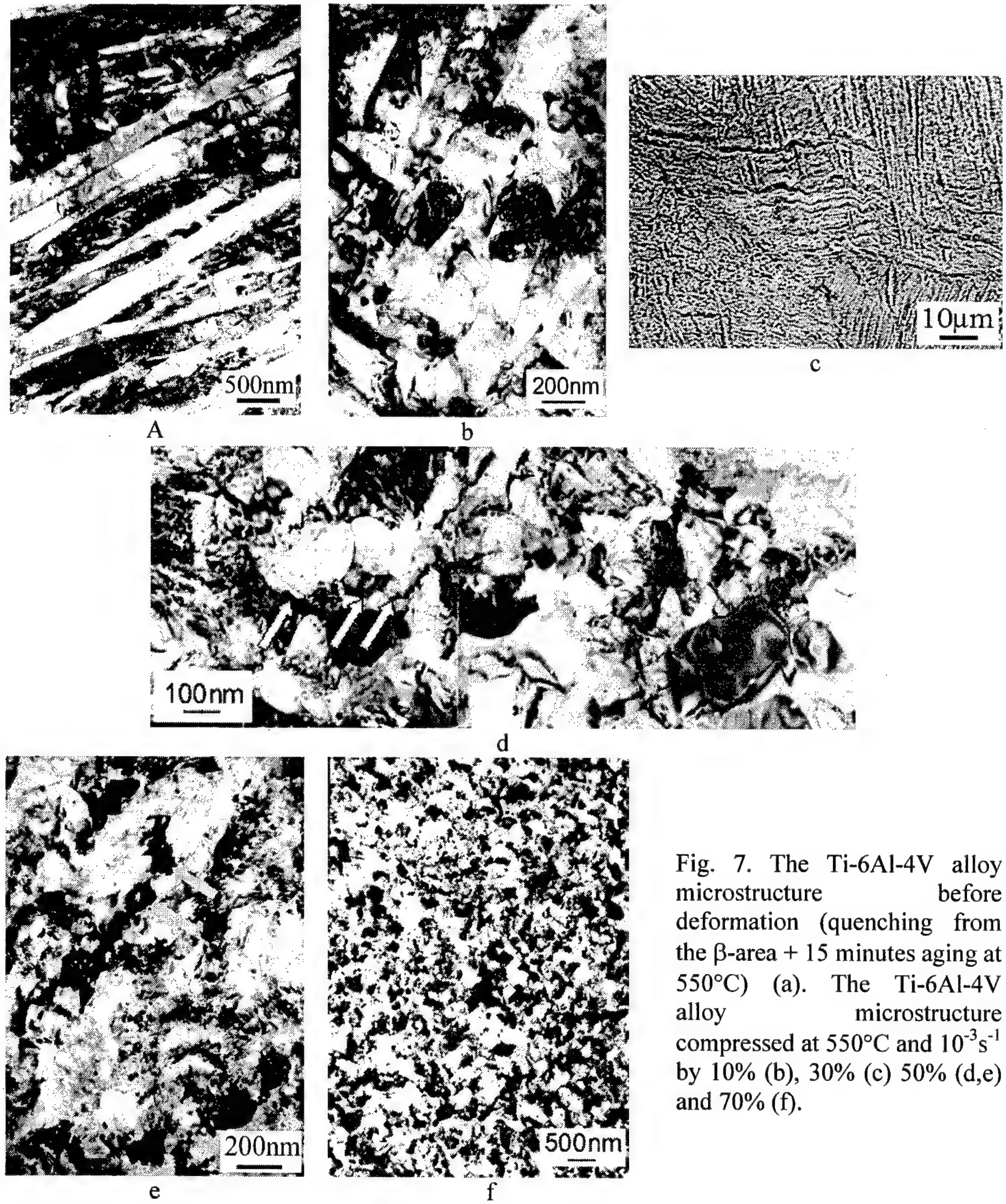


Fig. 7. The Ti-6Al-4V alloy microstructure before deformation (quenching from the β-area + 15 minutes aging at 550°C) (a). The Ti-6Al-4V alloy microstructure compressed at 550°C and $10^{-3}s^{-1}$ by 10% (b), 30% (c) 50% (d,e) and 70% (f).

After ε=70% deformation, a homogeneous structure with the globular grains of α- and β-phases is formed. The average grain size is approximately 0.2 μm. The SMC microstructure is characterized by the presence of a considerable number of fringe diffraction contrast, which is the evidence of the elastic lattice distortion, as well as of higher dislocation density in most of the grains. (Fig. 7f).

Thus, according to the results of microstructure research, the Ti-6Al-4V alloy microstructure transformation both at 800°C and 550°C occurs similarly, which proves the regularity character σ_s –

d for the alloy (Fig. 4). In the alloy at the initial deformation stage a deformation strengthening takes place owing to the increase in dislocation density in the phase plates. With the further growth ε in α- и β-phases transversal subboundaries are formed, which increase their disorientation up to high-angular interacting with lattice dislocations in the course of the further deformation. The plates turn in the direction of deformation.

According to some researchers [13, 19], to transform a lamellar structure into an equiaxial one in the two-phase titanium alloys, the reorganization of the semi coherent interphase boundaries into the non-coherent ones is necessary. It is known [38], that the semi coherent structure of the α/β boundaries in titanium (α+β)-alloys with lamellar structure is determined by the orientation phase ratio, which is formed as a result of the β⇒α transformation on cooling from the β-area. The α/β boundaries interaction with lattice dislocations in the course of deformation leads to elimination of the initial phase orientation, as a result of which semi coherent interphase boundaries are transformed into non-coherent ones. At low deformation temperatures an increase in lattice spacing discrepancy of the α- и β-phases can also promote the loss of interphase boundaries coherency due to the change of their alloy component. In the Ti-6Al-4V alloy the maximum discrepancy takes place at 550°C [29].

As a result of the interphase and intergranular boundaries reorganization, the mass transfer is intensified and grooves are formed on the α-plates surface. The β-phase interlayers and the α-plates divided into fragments are spheroidized. Along their boundaries a shift occurs as a result of grain-boundary sliding development (Fig. 5). Due to its effect supplementary stresses occurs, which results in defect density growth, diffusion activation and globularization escalation. The results obtained show that at the final deformation stage at T=550°C forming the SMC structure will lead to superplastic flow development. However, evaluating the coefficient of strain-rate sensitivity of the flow stress *m* on sample setting is problematic due to the low active deformation volume. Therefore, the present experiment was conducted in the course of successive compression of the prismatic samples along the three orthogonal directions.

A-1.3. Structural change and mechanical behavior of the Ti-6Al-4V alloy in the course of severe plastic deformations.

Unlike such well-known intensive plastic deformation methods as ECAE pressing and torsion under pressure, considerable strain of material can be achieved through successive samples pressing along the lines of three orthogonal directions. Such a deformation method is often called an 'abc' deformation (Fig. 8). With the aim of defining the possibility of applying this method for microstructure decomposition in bulk semi-manufactured products of the Ti-6Al-4V alloy, the laboratory testing of prismatic samples was undertaken. The previously quenched from the β-area samples with the initial dimensions of 16×18×20 mm were used. Before every turn the prismatic shape was restored by cutting the twisted surfaces. The microstructure evolution peculiarities and mechanical behavior of the alloy at 550 and 800°C were studied. The initial deformation strain rate and true strain at every stage were $10^{-3}s^{-1}$ and approximately 0.4 accordingly. The true strain was calculated as $\ln(h_0/h)$, where h_0 and h are initial and final height accordingly.

The deformation curves S-Σe, plotted for the 'abc' deformation of the Ti-6Al-4V alloy at 550 and 800°C and $10^{-3}s^{-1}$ are shown in Figure 9. The true curves S-e for both temperatures at early 'abc' deformation stage have a peak followed by the softening. After deformation at 550°C by the amount of Σe>1 the stage of the steady flow stage is noted with the true curves, whose extension increases with every loading step (Fig. 9a). After deformation at 800°C by practically the same amount of Σe>1, the shape of the curves changes as well, but they display noticeable strengthening when deformation increases (Fig. 9b). The resultant curves S-Σe have similar shape for both temperatures: a peak, softening and the steady flow stage, which shows that the average level of flow stress decreases with every loading step and arrives to practically constant value.

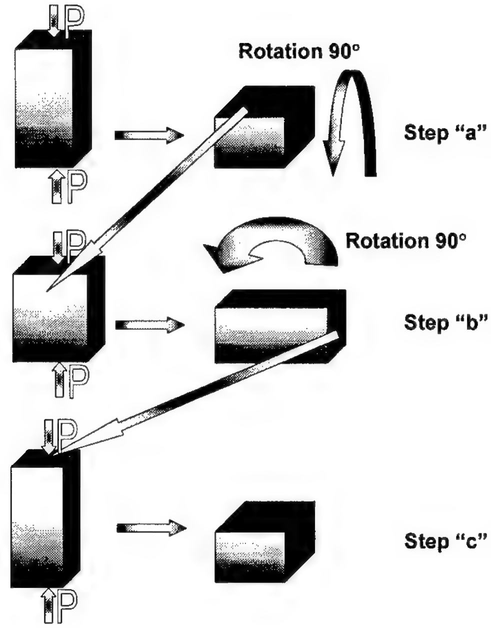


Fig. 8. Scheme of 'abc' deformation.

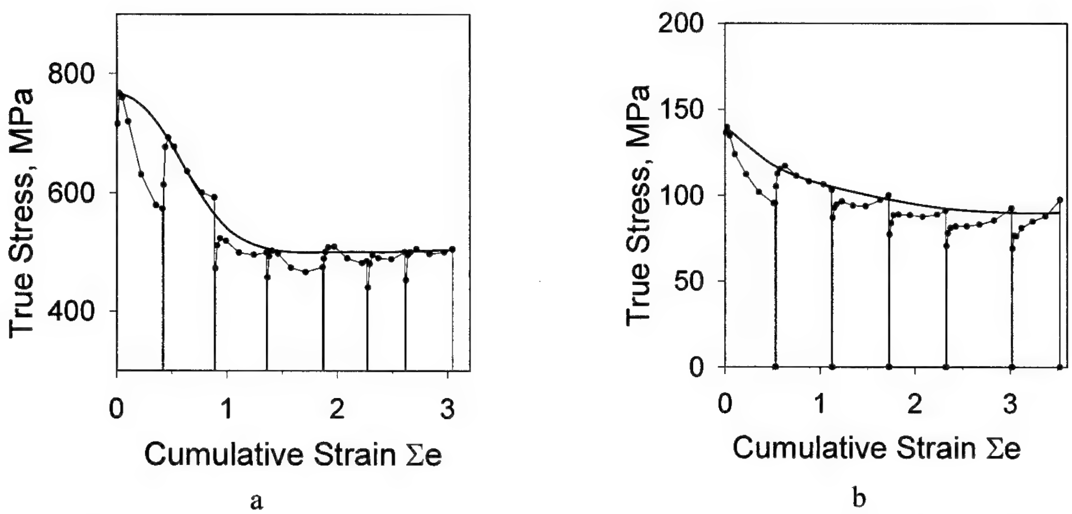
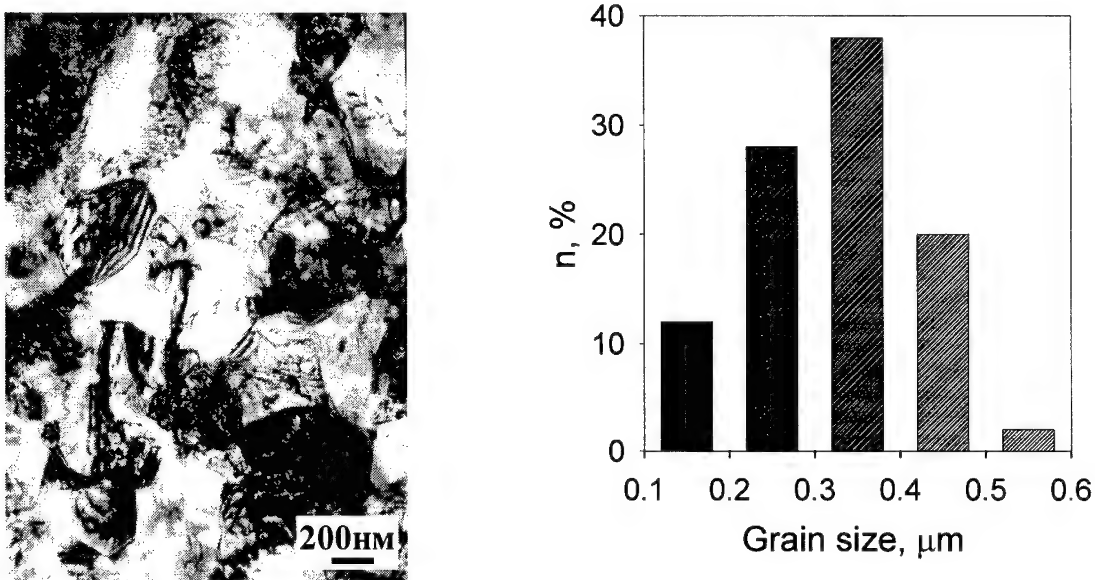


Fig. 9. Cumulative curve S-Σe for 'abc' deformation at 550°C (a) and 800°C (b), and strain rate $10^{-3}s^{-1}$ of Ti-6Al-4V alloy, quenched in water from β-area.

It is worth mentioning that while analyzing the deformation curves one has to take into account such factors as texture change (first and foremost, a metallographic one at initial stages of deformation) and possible microstructure change in the course of sample heating and cooling acts. During the first compression deformation a primary turn of the α-plates in a sample takes place in the direction perpendicular to the deformation axis. On turning of the samples (Fig. 8), they appear to be oriented mainly parallel to the deformation axis. This results in flow stress increase at the second step (which is displayed by the curves at both temperatures). However, with the deformation accumulation and globularization processes development (Fig. 6e, 7f) the texture influence decreases, that is why after two-three steps the flow stress of the end of the previous step and of the beginning of the following step become close. Apparently, the softening on heating up to the deformation temperature is to appear most dramatically at high temperatures (800°C) and to be manifested in decreasing the

flow stress at every following stage. Evidently, it is this very factor that is responsible for the difference in flow stress between the end of the previous step and the beginning of the following step at 800°C.

Analyzing microstructure of the sample compressed at T=550°C up to $\Sigma e=3$ showed that it is homogeneous in all its volume with the grain size of 0.4 μm (Fig. 10a, b). The bar chart of the grains distribution by their size indicates the absence of grains more than 0.6 μm in size. The lower strains preserved some parts with lamellar phase structure.



a б

Fig. 10. The microstructure (a) and the grains distribution by size (b) in the Ti-6Al-4V alloy after the 'abc' deformation up to $\Sigma e=3$ at 550°C and $10^{-3}s^{-1}$.

The presence of the steady flow stage on true deformation curves is evident of the alloy transformation into superplastic flow in the course of 'abc' deformation. As the globular structure is formed in the total sample volume, this enables us to evaluate the coefficient of strain rate sensitivity of the flow stress *m* and to determine the activation energy at this deformation stage. To determine the m coefficient the prismatic samples of 7×7×10 mm were tested in the range of deformation strain rate of 10^{-4}-10^{-2} s^{-1} at 550 and 800°C with e=0.4 и $\Sigma e=0.9$. The calculations were made out of inclination curves σ-ε. At the temperature of 550°C and strain rate of $10^{-3}s^{-1}$ the following m coefficient values were obtained: 0.24 for e=0.4 и 0.42 for $\Sigma e=0.9$. At the steady flow stage the value of the apparent deformation activation energy for the temperature range of 525-600°C equaled 185 kJ/mole, and within the temperature range of 775-825°C the similar value of 210 kJ/mole was obtained. The calculated values practically coincide with the ones that take place during the superplastic deformation of the two-phased titanium Ti-6Al-4V alloy (216 kJ/mole for 925°C). [39]. These data display the important role of superplastic deformation mode in forming SMC structures in two-phase Ti-6Al-4V titanium alloys.

Thus, for obtaining the SMC structure with the grain size of less than 0,5 μm, which is required by the project, the preferred deformation mode is T=550°C and deformation strain rate of $10^{-3}s^{-1}$. As the critical deformations before the first globularization signs rise dramatically with temperature decreasing, it is necessary to have the strain of not less than $\Sigma e=3$ to form the SMC structures within the whole sample volume. Moreover, as it follows from the experiments conducted to provide homogeneous microstructure, all the structural and technological factors promoting the process activation should be taken into account. The deformation scheme development which could ensure the even deformation distribution is also of importance. The following chapters of this report are devoted to solving the above-mentioned problems.

A-2 Task. Investigation the influence of the temperature-strain rate deformation mode and initial microstructure (grain size, volume fraction of phase, α-phase plates thickness) on formation kinetics of the SMC structure, as well as on its homogeneity, cavitation and mechanical properties.

A-2.1. Investigating the influence of initial microstructure on the globularized grain size and their volume fraction.

Type of the initial microstructure of two-phase titanium alloys is one of the strongest factors that significantly influence the refining kinetics of the microstructure as well as on its homogeneity [21, 23] and on billets workability [37]. The latter is of paramount importance, as the decrease in processing temperature down to the temperatures of SMC structure forming leads to decrease in material ductility and, consequently, restricts attainment of the required strains. Furthermore, the microstructure tends to be heterogeneous and combines areas of lamellar and globular components in large scale titanium alloy bars (which often are exposed to forging in the β-area to improve their microstructure). After heat treatment in the β-area, the microstructure can be resulted in α′-martensite or (α+β)-lamellar microstructure depending on cooling strain rate. As it has already been stated above, the former is preferable, as it allows the most fine lamellar (α+β) microstructure in the course of dissociation. However, the heat treatment in the β-area inevitably leads to grain coarsening and, consequently, to ductility declining.

In this connection the influence of various types of the Ti-6Al-4V alloy initial microstructure on SMC structure parameters forming in the process of deformation was researched. The following microstructure types were chosen: α′-martensite microstructure (after quenching from the β-area, the heat temperature of 1010°C, as is in A-1.1. section), (α+β)-lamellar microstructure (after cooling in the air from the β-area, the heat temperature of 1010°C), globular microstructure (formed by means of multiple isothermal forging at 700°C), and "bi-modal" microstructure (after (α+β) multiple isothermal forging at 950°C followed by cooling in the air), which are Conditions **A, B, C** and **D** accordingly.

The cylindrical samples of ∅10×15 mm were deformed by 70% compression at 550 °C and at the strain rate of $10^{-3}s^{-1}$. The microstructure was researched at the section parallel to the compression axis in the sample centre.

Initially, the course-lamellar structure consisted of α-phase plates colonies approximately 0.5 μm thick with thin (about 50 nm) β-phase interlayer (Fig. 11a). Along the boundaries of the initial β-grains the α-case approximately 15 μm thick was noted. The globular structure is characterized by the α- and β-phase particles of about 0.8 μm in size. Both particles with increased dislocation density and dislocation-free particles are found. Along the boundaries of some particles the thickness extinction contours are present (Fig. 11c). The "bi-modal" microstructure before deformation was by the initial globular α- phase particles sized 19 μm, as well as by the β-transformed grain areas with secondary α- phase plates about 1 μm thick (Fig. 11e). In some grains a substructure was found.

The stress-strain curves for Ti-6Al-4V alloy deformed at 550 °C and the strain rate of $10^{-3}s^{-1}$ in various conditions are shown in Figure 12. It can be seen that for all the four conditions a flow stress peak followed by softening found on a σ-ε curve. The maximum flow stress value (650MPa), reached earlier (3%) than in the other conditions (6-12%), is characteristic of the alloy with initial martensite structure. The same condition is characterized by significantly more intensive softening as compared to the coarse-lamellar structure. The transformation to microstructures of the globular type considerably decreases the flow stress and softening intensity is reduced. The least flow stress level is displayed by the alloy with globular microstructure forged at 700°C. The results, in accordance with publication data [13, 24, 37], show the higher tendency of coarse-grained microstructures to localize deformations.

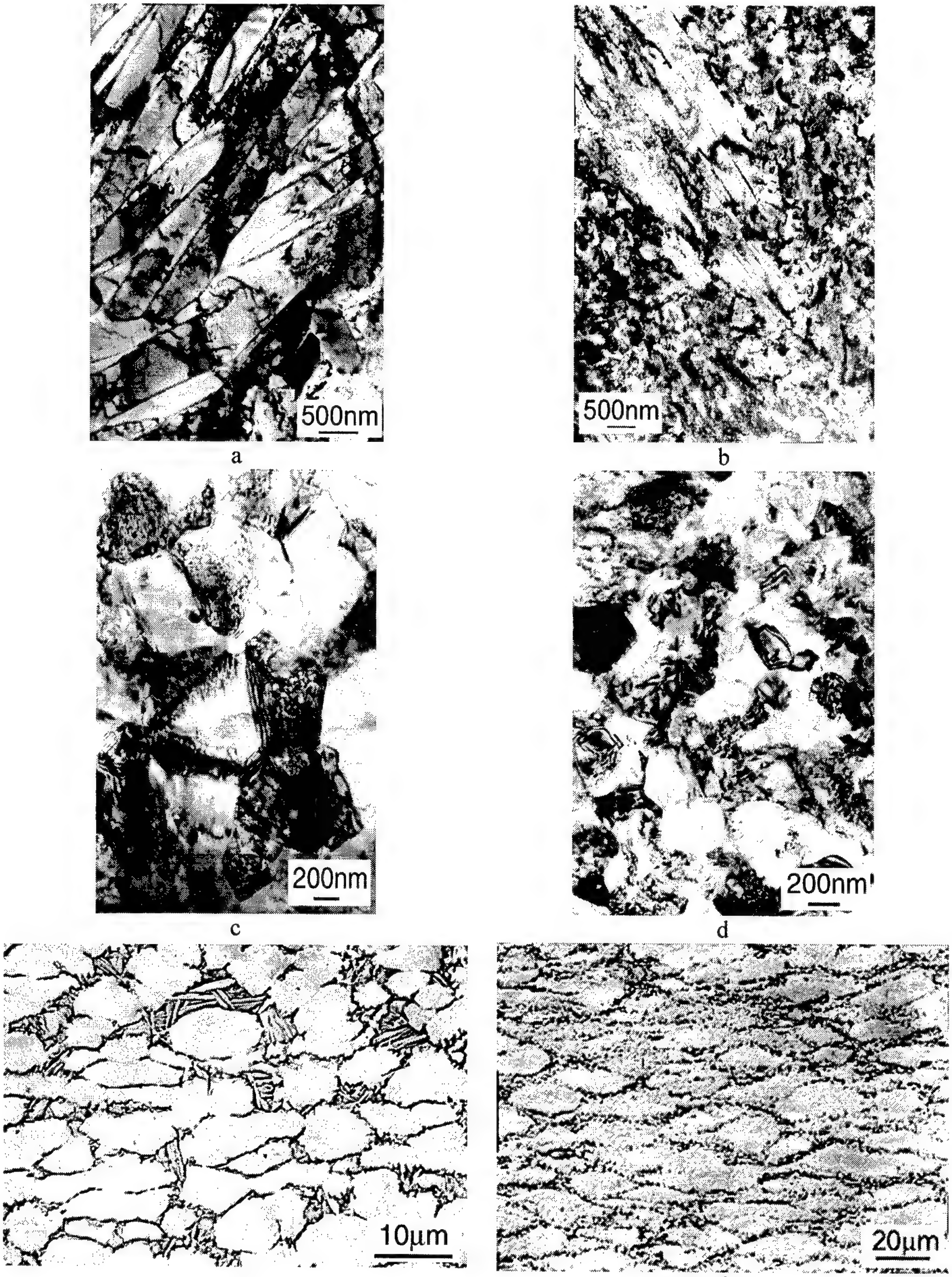


Fig. 11. The Ti-6Al-4V alloy microstructure before deformation (a, c, e) and after 70% compression deformation (b, d, f) at 550 °C and at the strain rate of $10^{-3}s^{-1}$. Initial conditions: (after cooling in the air from the β-area) (Condition B) (a); globular microstructure (Condition C) (b); "bi-modal" microstructure (Condition D) (c).

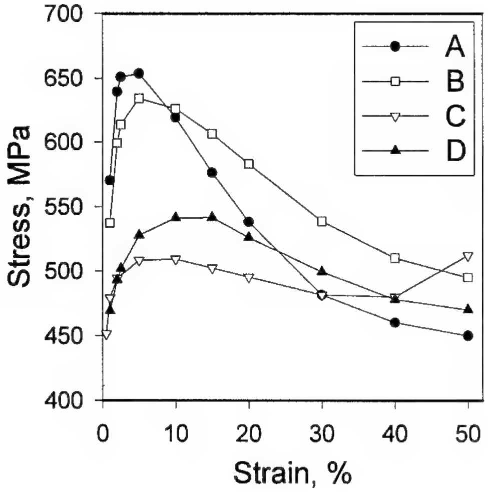


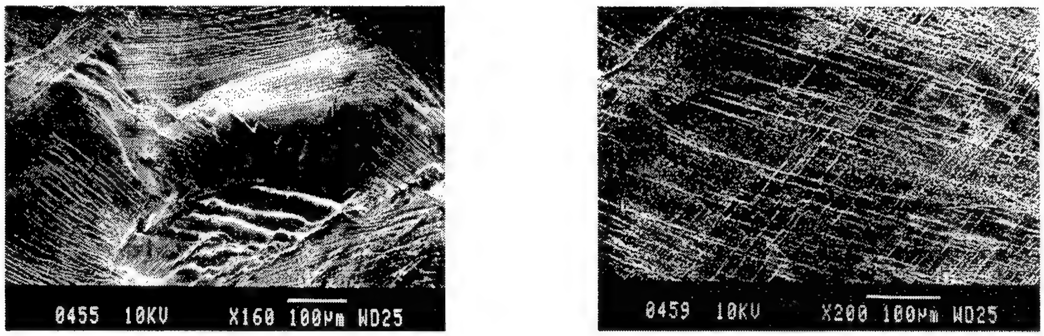
Fig. 12. The flow stress-strain curves of the Ti-6Al-4V alloy deformation at 550 °C and strain rate of $10^{-3}s^{-1}$ with the initial martensite structure (quenching from the β-area) (Condition **A**); rough-lamellar structure (cooling in the air from the β-area) (Condition **B**); globular structure (formed by means of multiple isothermal forging at 700°C) (Condition **C**); and "bi-modal" structure (after (α+β) multiple isothermal forging at 950°C followed by cooling in the air) (Condition **D**).

After 70% compression deformation of alloy with initial martensite and globular microstructures (Conditions A and C), a homogeneous globular SMC structure (Figs. 7d and 11d) with the grain size of about 0.3 μm is formed. As a result of deformation, SMC grains of about 0.3 μm are formed in the samples with coarse-lamellar structure (Condition B) as well. However, the microstructure is heterogeneous – up to 30% of the lamellar component is preserved (Fig. 11b). In "bi-modal" microstructure as a result of deformation it is only the lamellar component that is transformed to form the SMC size grains. Simultaneously, the α-globules are only slightly extended in the direction of metal flow (Fig. 11f).

Apparently, the main reason of such an influence of the initial microstructure type on its transformation in the course of deformation is determined by the difference in the plastic flow homogeneity. When martensite and coarse-plate conditions are compared, this has been clearly demonstrated by examining the sliding traces character. The deformational relief was researched after deformation of the prismatic samples sized 7×7×10 mm until ε=15% at 550 °C and at $10^{-3}s^{-1}$. To do so, the sample lateral surface, which was previously polished electrolytic to prevent oxidation, was covered with boric acid at 200°C, which was removed with water when the deformation stopped. Comparing deformation relief on the sample surface of these conditions shows significantly more homogeneous character of forming sliding traces in the martensite structure alloy (Fig. 13). Fine-lamellar structures formation during the martensite decomposition when heating up to deformation temperature and absence of the α-case result in a more homogeneous deformation course in the alloy. In case of "bi-modal" structure, the recovery processes are more intensive in the α-phase globules then in the lamellar component. The strength of the above components is about the same for the temperature range under research [38]. Because of this, the defects accumulation, promoted by the dislocation deceleration by the α-phase particles, results in globularization development combined by forming SMC of the grains size. The plastic flow localizes in this component with the SMC grains, which decelerates the α-phase globules recrystallization. At the same time, in case of the microstructure previously forged at 700°C, the deformation at 550°C leads to dynamic recrystallization development, both in the α-phase and β-phase grains. The above is testified by the fact of forming grains of considerably smaller size. During this the globular microstructure provides more homogeneous plastic flow as compared to the "bi-modal" one due to the absence of lamellar component and smaller size of the α-phase globules.

Thus, it is preferable for SMC structure forming to make use of the alloy conditions with the initial martensite structure or of the globular type microstructure, in which β-transformed components areas are absent. The latter implies at least double-stage processing of the alloy: at the first stage it is forging a billet with the initial martensite structure at, say, 700°C to obtain a globular

microstructure. The globular microstructure allows us to significantly increase the alloy ductility as compared to initial coarse-grained structure at the second stage of processing, executed at low temperatures to form SMC structures [40]. As far as process economy is concerned, the forging option is undoubtedly preferred, which again should be executed at low temperatures.



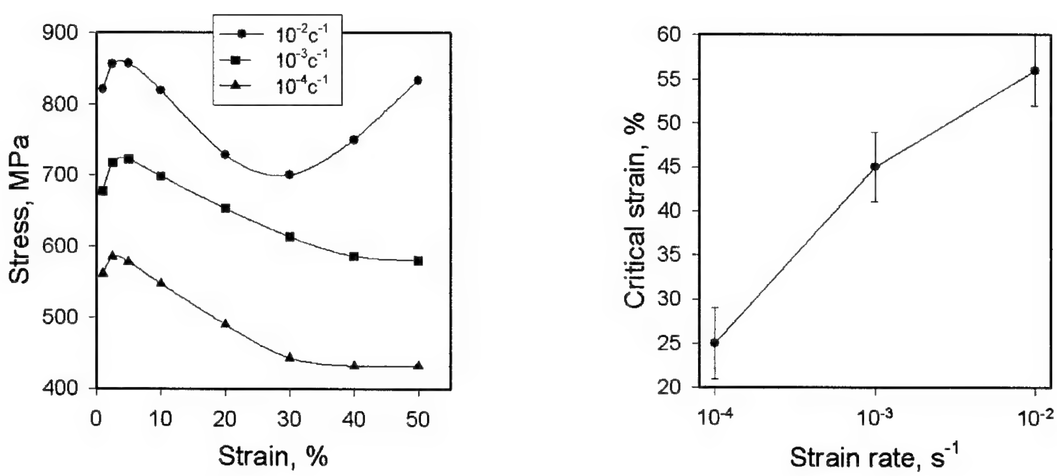
a б

Fig. 13. Deformation relief of the samples with initially fine lamellar structure (after cooling in the air from the β-area) (a) and martensite (after quenching from the β-area) (b) with microstructures after deformation at 550°C and $10^{-3}s^{-1}$ by 15%.

A-2.2. Determining critical deformation for forming of the first globular grains in various temperature-strain rate deformation conditions.

In the course of SMC structure preparation in bulk semi-manufactured materials, the inevitable irregularity in distribution of strain and strain rates within a billet should be taken into account. Therefore, the areas, where globularization went on faster then in the other billet parts, may lead to deformation localization and structural irregularity. In this connection, the influence of the deformation strain rate on critical strain for forming the first globularized grains was investigated.

The cylindrical samples of ∅10×15 mm with the martensite structure (after quenching from the β-area) were exposed to deformation by 5, 10, 20, 40 and 60% at 550°C and at the strain rates of $10^{-2}s^{-1}$; $10^{-3}s^{-1}$; $10^{-4}s^{-1}$. To reveal critical deformation of the first globular grains formation in the central area of the compressed samples the microstructure was researched.



a b

Fig. 14. Stress-strain curves at different deformation strain rate (a); dependence of critical deformation of globular grains forming on deformation strain rate (b), obtained for the samples with the initial martensite structure (quenching from the β-area). Deformation temperature is 550°C.

The curves σ-ε plotted at 550°C for various deformation strain rates are shown in Figure 14a. On increasing the deformation strain rate by two orders of magnitude, the flow stress increase nearly half as much again. All the three curves are of the similar shape – with a peak followed by softening. However, at the strain rate of $10^{-2}s^{-1}$, starting with 30% deformation, repeated, strengthening is noted, which is unavailable at lower strain rates. With reference to Fig. 14b, it can be seen that lowering deformation strain rate results in decrease in critical deformation required for the first globular grains formation. When testing strain rate increases by two orders of magnitude, the critical deformation increases twice. The above result shows that, when choosing technological modes and processing schemes, it is of paramount importance to take into account the deformation and strain rate deformation distribution within a billet due to their significant influence on globularization kinetics.

A-3 Task. Investigation the initial microstructure influence on defects formation (wedge cracking and cavitation)and workability of the alloy.

A-3.1. Investigation of the influence of initial microstructure and temperature – strain rate conditions on defects formation (wedge cracking and cavitation) and ductility.

In the conditions of low temperature deformation, in which the SMC structure formation is expected, it is of paramount importance to determine acceptable strains before cracks and pores are formed. They greatly depend both on the initial alloy microstructure and on the temperature-strain rate deformation conditions. Such researches were conducted in a number of publications [41, 42, 43], but they are not known to the authors of this paper as far as the temperature range discussed in this paper is concerned. Usually the defects that arise during the heat deformation are subdivided into three types: formed 1. due to the microscopically irregular deformation in the interior of metal. 2. as a result of deformation localization caused by the different temperature of the billet surface and billet body and/or by the presence of a coarse-grain structure in it. 3. due to the microscopically irregular flow of the metal [37]. The former are of special interest for the present research as a basic defect type, which can take place during isothermal deformation at low temperatures. It is worth mentioning that the second defect type was not found in deformation modes used in the experiment.

For investigating the three alloy conditions were used: with martensite, coarse-lamellar and globular microstructures (Conditions A, B and C accordingly). The modes for the alloy conditions obtaining and the microstructure parameters are discussed in the A-2.1. Section. The samples to be researched with the test portion sized ∅5×25 mm were manufactured to be tested for stretching. Choosing this samples testing scheme is determined by the fact that the stretching tensions are responsible for forming cracks and pores on the billet surface. Testing was conducted at the deformation temperatures of 550, 650 and 800°C and at the strain rate of $10^{-3}s^{-1}$. The indexes of ductility were determined by aspect ratio (AR) δ and reduction area (RA) ψ. Compressed samples were used to research cavitation. To do so, a compressed sample section was cut along the deformation axis after which its mechanically polished surface was studied with the help of an optical microscope.

The table 4.1.1 shows relative lengthening and relative narrowing values of the alloy in three conditions at 550, 650 и 800°C. It can be noticed that lowering temperature decreases both (AR) and (RA) of the alloy. However, the material condition greatly influences this change. The greatest plastic characteristics are noted in the SMC condition. It is important that in condition A the indexes δ and especially ψ are higher than in condition B. Forming more fine-lamellar microstructure promotes the alloy ductility, especially at low deformation temperatures. It is worth mentioning that in condition A the flow stresses at the initial deformation stage were somewhat higher then in condition B. The least flow stress was revealed in the condition with the globular SMC structure (condition C). The data for 550°C are shown in the Figure. 2a. Thus, conditions A and B are characterized by better workability as compared to condition C.

Table 4.1.1

Temperature and deformation strain rate influence on plastic characteristics of the Ti-6Al-4V alloy with different initial structure.

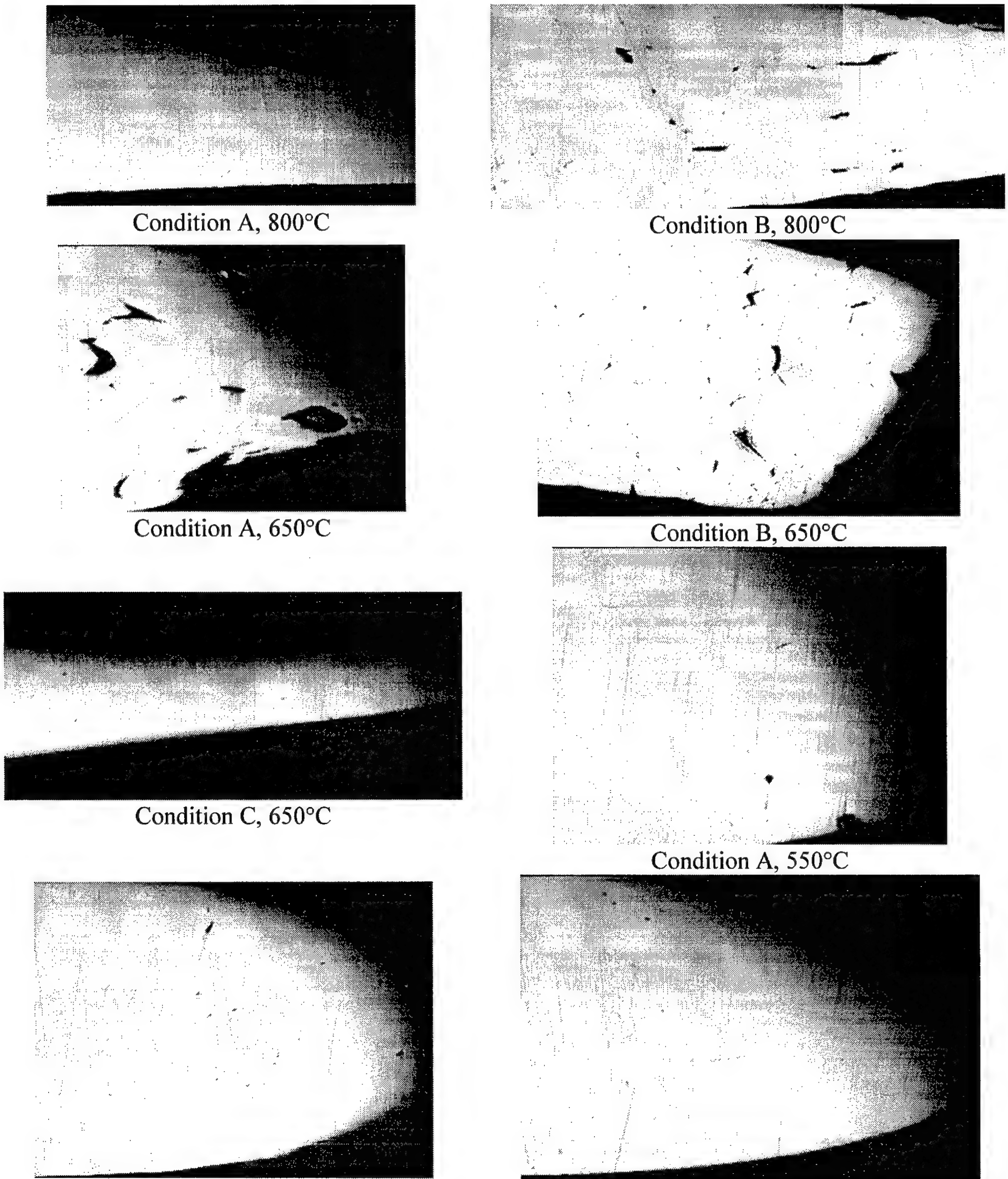
Condition of the alloy	Strain rate, s^{-1}	δ, %			ψ, %		
		550°C	650°C	800°C	550°C	650°C	800°C
A	10^{-2}	30	32		96	92	
	10^{-3}	46	60	120	79	99	99
	10^{-4}	63	97		99	99	
B	10^{-2}	22	21		73	71	
	10^{-3}	33	53	100	77	89	99
	10^{-4}	37	78		80	98	
C	10^{-2}	30	126		96	99	
	10^{-3}	96	200	>700	99	99	99
	10^{-4}	217	528		99	99	

Microstructural research of the sample neck areas revealed two defect types: wedge-shaped cracks and pores (Fig. 15). The wedge-shaped cracks were formed in the coarse-grain alloy conditions in triple joints of the grains boundary when lowering the deformation temperature down to 650°C. However, at 550°C the total fraction of cracks and pores decreased, just as in the case of increasing deformation strain rate (Table 4.1.2). Reducing cavitation is caused by reducing the plastic deformation value of the samples with lowering the testing temperature. On comparing coarse-grain conditions no significant difference in defect fraction is found. However, it is much greater than in the alloy with the SMC structure. At decreasing of deformation temperatures probability of defects formation increase due to macrolocalization of shear deformation [37]. The latter leads to (for instance at compression) formation of shear localization [37]. This circumstance demand of strain limitation during processing in order to avoid failure of the billet.

Tabl. 4.1.2.

Temperature and deformation strain rate influence on cavity volume fraction of the Ti-6Al-4V alloy with different initial structure.

Condition of the alloy	Strain rate, s^{-1}	Total fraction of cracks and pores, %		
		550°C	650°C	800°C
A	10^{-2}	0,45	3,1	
	10^{-3}	0,2	3,71	0
	10^{-4}	2,13	2,92	
B	10^{-2}	1,1	0,98	
	10^{-3}	0,27	2,72	1,72
	10^{-4}	2,56	2,3	
C	10^{-2}	0,27	0	
	10^{-3}	0,13	0,14	0
	10^{-4}	0	0,2	



Condition A, 800°C

Condition B, 800°C

Condition A, 650°C

Condition B, 650°C

Condition C, 650°C

Condition A, 550°C

Condition B, 550°C

Condition C, 550°C

Fig. 15 Temperature and initial structure influence on crack formation and cavitation of the Ti-6Al-4V alloy samples after tension test at $10^{-3}s^{-1}$.

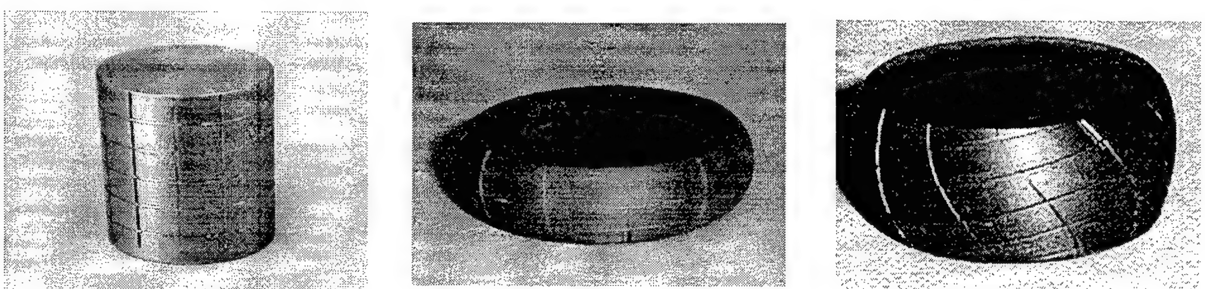
Thus, the results obtained show that to prepare the SMC structures in the alloy it is preferable to use the alloy condition with the martensite structure, which is somewhat more plastic at 550°C than the alloy with the coarse-lamellar structure. As a result of the SMC condition formation, the alloy deformability also improves within the temperature range under research in the process of structure preparation. As cavitation develops more intensively on a surface, which is exposed to tensile stress, the role of this factor should be evaluated in an instance close to real billet deformation case. This task was discussed issuing from the example of sample compression of the

samples with different initial microstructure at various deformation temperatures (see A-3.2. section).

A-3.2. Determination of allowable strains during forging.

Cylindrical samples 20 mm in diameter and 30 mm in length, having initial martensite (water quenching from β-region) (Condition **A**); coarse lamellar (air cooling from β-region) (Condition **B**) and globular (obtained by multiple step isothermal forging at 700°C) (Condition **C**) structures with the coordinate scale laid out on the generatrix surface (Fig. 16a) were compressed by 30, 50 and 70% at temperatures 500, 550, 650°C and the strain rate 10^{-3}sec^{-1}. The dimensions of coordinate scale cells were 5×5 mm. The true strain values in various zones of the sample were determined by a change in cell dimensions. It should be noted that because of instability of deformation, especially at 550°C, the scale underwent significant distortions (Fig. 16c).

The surface analysis has shown that in Condition B cracks were formed already at the temperature 650°C in the scale cell in which the strain value was 85%, and at 550°C at the strain value 50%. Whereas in other Conditions surface cracks were observed at larger strain values. The more fine lamellar structure (Condition A) essentially increases ductile parameters and formation of small surface cracks occurs at the temperature 550°C and the strain value equal to 55%. No surface cracks are formed on the surface of the samples in Condition C both at the temperatures 550 and 650°C at maximum strains in scale cells equal to 65 and 85%, respectively, and in the case of the deformation temperature decreased to 500°C and deformation equal to 45%. On the whole, the results obtained fit well with the data of tensile tests. On the basis of the data obtained one can determine allowable strains for multiple isothermal forging. For processing of the alloy with initial martensite structure at the temperature 550°C the allowable strains until cracks occurrence should not exceed 55%, which can be reached at the deformation no more than 50%.



a b c

Fig. 16. Appearance of Ti-6Al-4V alloy samples before (a) and after deformation at strain rate 10^{-3}s^{-1} and temperature 650°C in condition A with initially martensite (b) and at temperature 550°C in condition C with globular microstructure (c).

Activity A-4. Computer modeling of multiple isothermal forging for manufacturing tooling providing production of billets, ∅150×200 mm with uniform microstructure and a grain size less than 0.5 microns.

A-4.1. Computer modeling and manufacture of tooling providing an increase in technological ductility and uniformity of stress and strain distribution in each stage.

The results obtained while fulfilling Activities A-1, A-2, A-3 are taken into account as input data for modeling. In particular, the flow stress-strain curves for the alloys To-6Al-4V with initial martensite structure obtained at T=550°C and the strain rate 10^{-3}s^{-1}. At the same time, as shown in Activity A-1 in the process of deformation of the sample with a turn performed successfully in three orthogonal directions the initial lamellar microstructure transforms to the SMC globular one.

Concurrently the mechanical behavior of the alloy changes essentially. If in the in the initial condition with the mareteniste structure the curve has a peak followed by softening (Fig. 2a) then in the SMC condition at the final stage of deformation there takes place a steady flow almost on all the curve (Fig. 9b). Another important fact is that in the coarse-grained condition the allowable strains until occurrence of cracks on the lateral surface of the billet should not exceed 55%. These values are achieved at high deformation no more than 50%. In this connection, while selecting the method of forging of billets with their successive turn in three orthogonal directions it is necessary to limit (restrict) the strains of high deformation , especially, in the first stages of loading. That is why the value of high strain equal to 50% (the strain value of 50%) was taken for modeling

The center of deformation which prevents premature initiation of cracks on the lateral surface was created by the specially developed device in which deformation is realized via compression by height. The device promotes plastic flow of the material in one direction which does not coincide with the direction of deforming load [44]. The die set according to the US Patent (4721537, 26.01.1988) is a close version of our device but it promotes plastic flow in two directions. So, the following input data were taken for numerical modeling of multiple step forging:

-Two billets with different initial microstructures in the form of the parallelepiped with the side dimensions 133x133x220 mm are deformed under isothermal conditions.

-Due to symmetry of the billet, ½ portion of the billet is subjected to modeling.

-The strain rate of the billet is determined by the velocity of displacement of the upper movable punch and is equal to 0,50 mm/sec.

- The friction coefficient by Ziebel is taken constant and equal to 0.3 that corresponds to the deformation at T=550°C.

-Microstructure in the initial stage of deformation is coarse-grained of a martensite type Microstructure in the final stage – SMC of a globular type with a grain size less than 0,5 μm.

-Material of the billet – alloy Ti-6Al-4V.

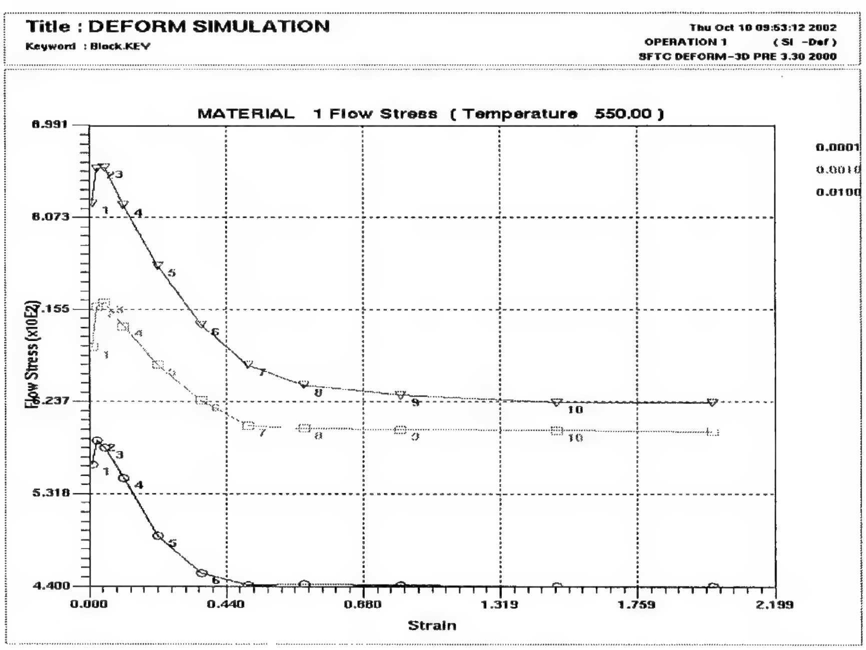


Fig. 17. True stress – true strain curves of alloy with initially coarse grain martensite structure at 550°C and at strain rates 10^{-4}, 10^{-3}, 10^{-2} s^{-1}.

Properties of the material are determined by the model of a plastic body described by curves shown in Figs. 17 and 18. Curves shown in Fig. 17 describe deformation behavior of the material of the billet with initial coarse-grained lamellar structure. Curves shown in Fig. 18 describe deformation behavior of the material of the billet with initial SMC structure.

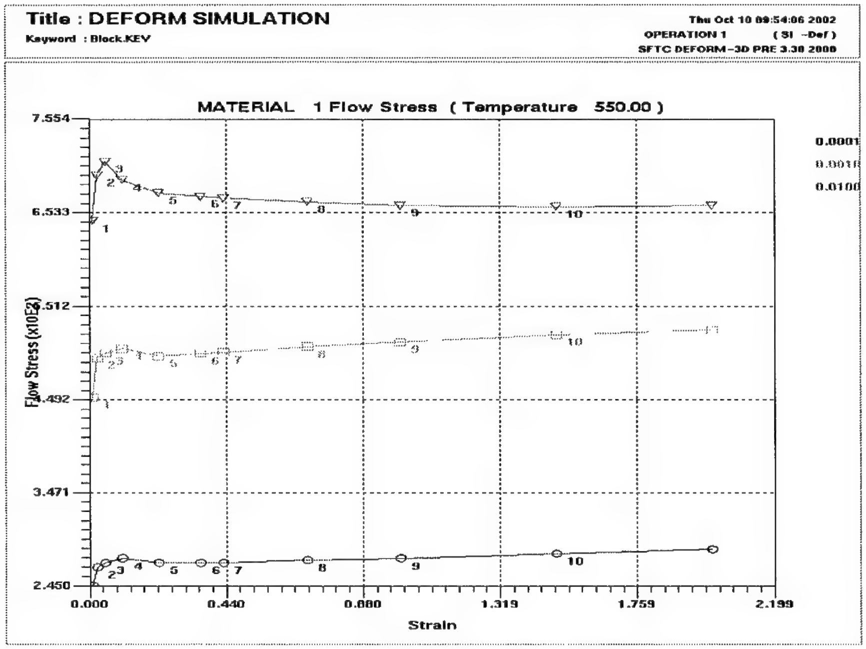


Fig. 18. True stress – true strain curves of alloy with initially SMC structure at 550°C and at strain rates 10^{-4}, 10^{-3}, 10^{-2} s^{-1}.

4.1.1. Stress and strain states of the billet out of the titanium alloy Ti-6Al-4V with initial coarse-grained martensite structure subjected to deformation with plastic metal flow in one direction.

Figures 19, 20, 21, 22 show, respectively, the initial position of the billet and distributions of effective strains, stresses and rates after compression of the billet from 220.0 mm to 133.0 by the upper movable punch. It is seen that the similar position of the billet in the cavity of the die set causes the occurrence of one band of shear strain. As the specified strain value is achieved it results in formation of a fold that is inadmissible during strain processing. In view of a significant difference in strains between different portions of the billet in the process of following deformation with a turn over 90° this will lead to preferable deformation in this band.

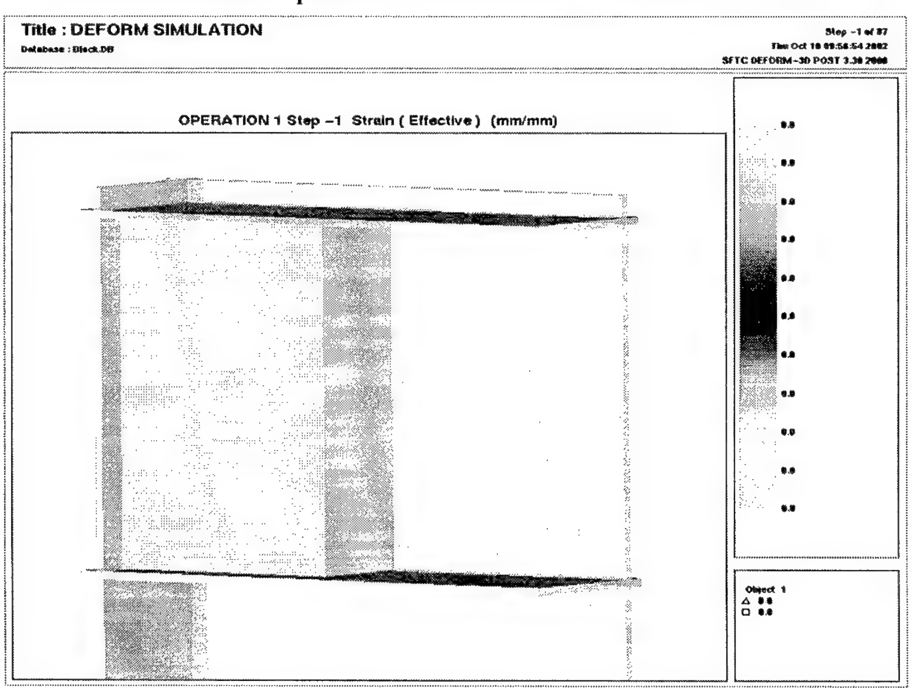
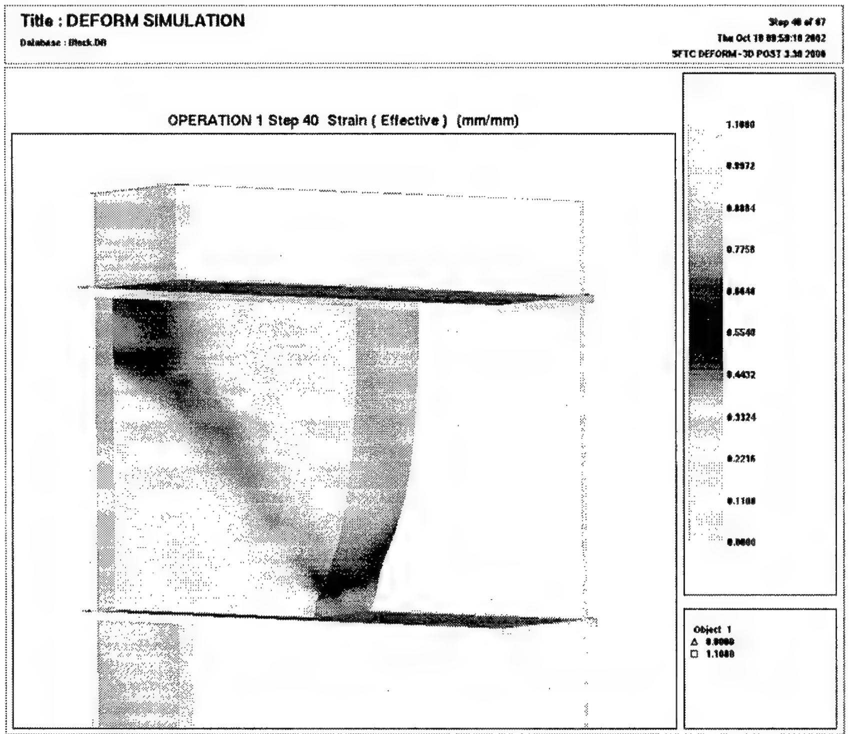
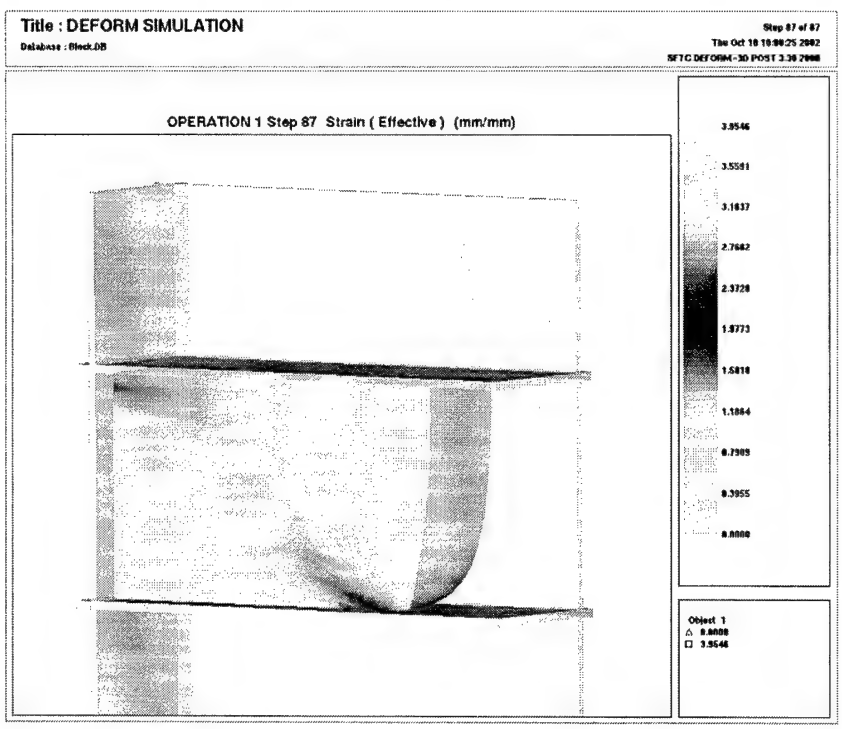


Fig. 19. Initial position of the billet

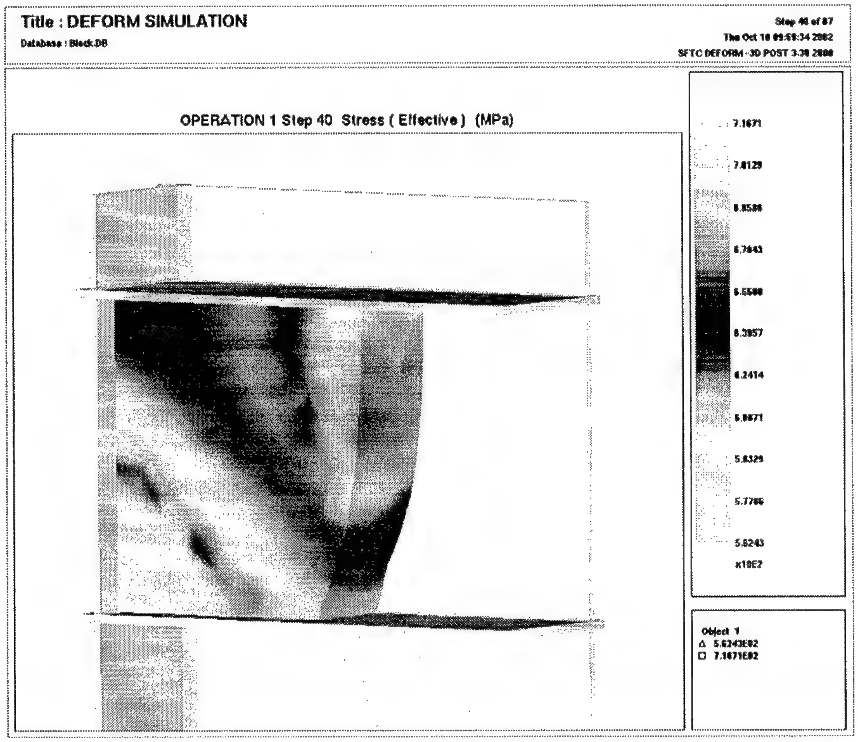


a. Distribution of effective strain after displacement of the movable punch by 40.0 mm

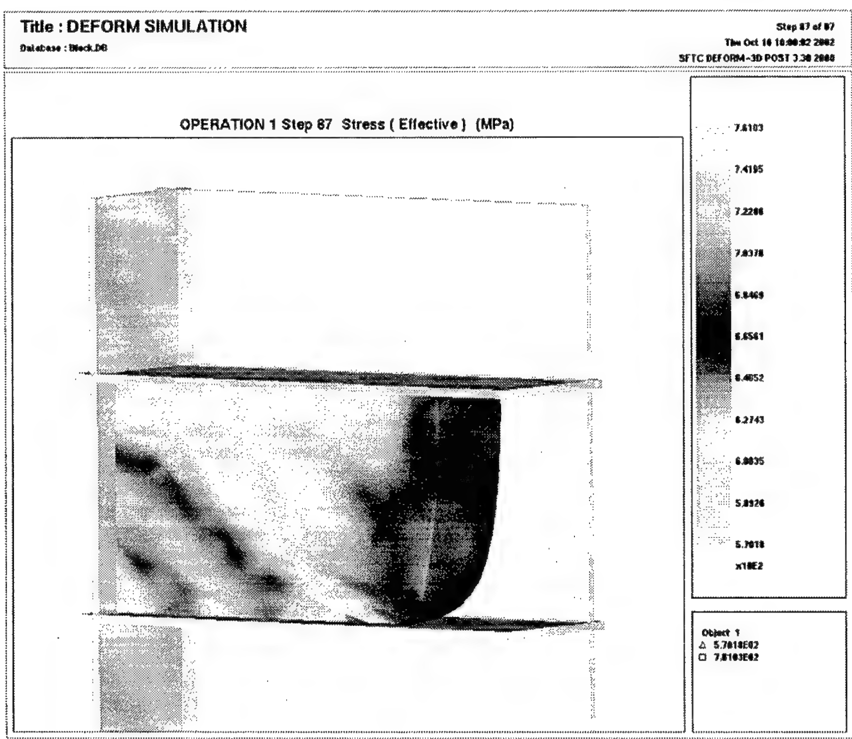


b. Distribution of effective strain after displacement of the movable punch by 87.0 mm

Fig. 20. Distribution of effective strain after compression of the billet by the upper movable punch at the velocity 0.5 mm/sec with the height 220.0 mm to the height 133.0 mm.

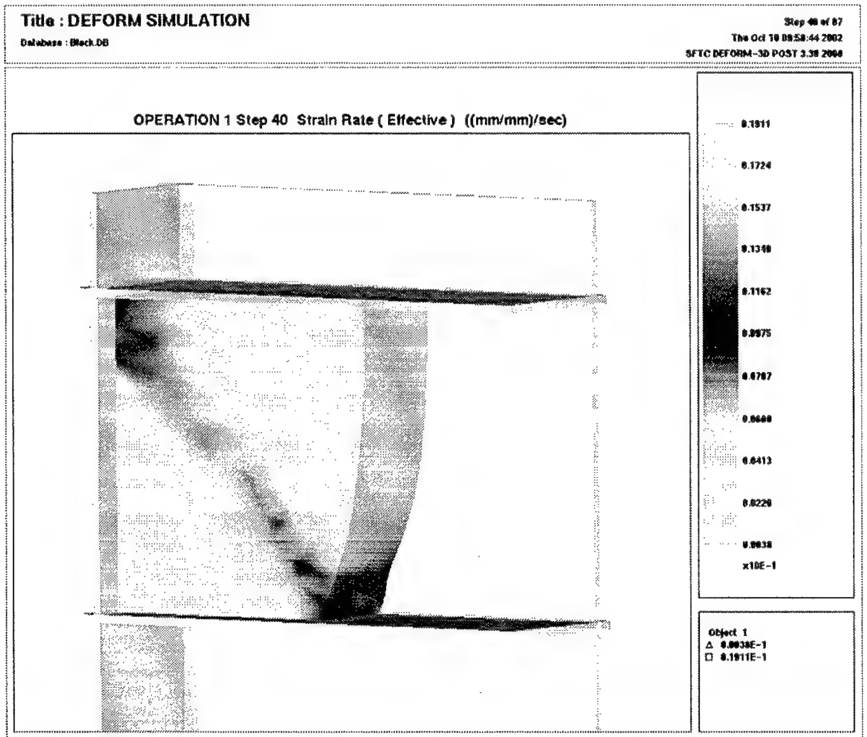


a. Distribution of effective stresses after displacement of the movable punch by 40.0 mm.

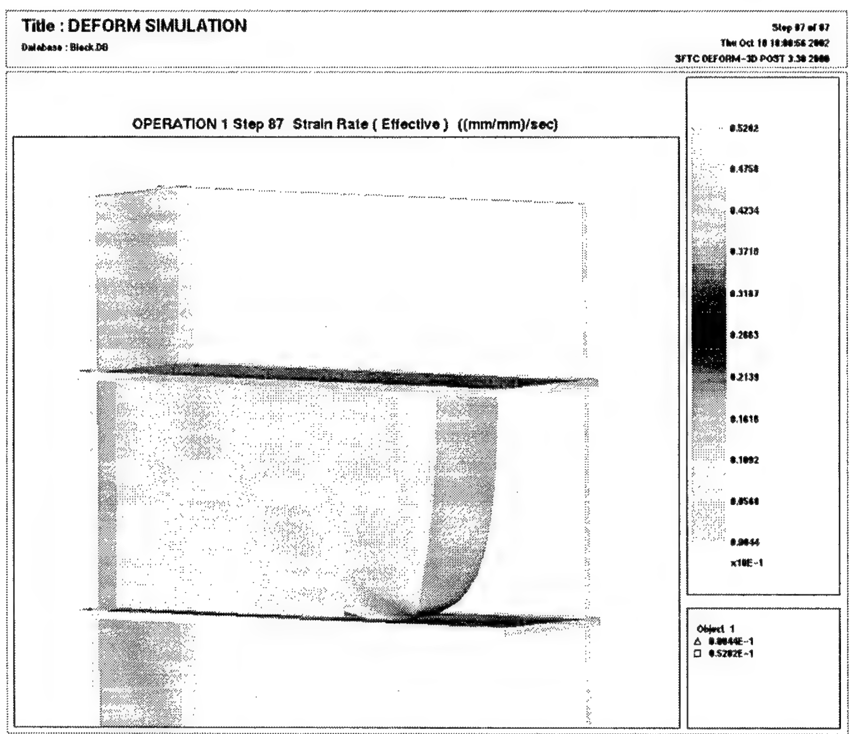


b. Distribution of effective stresses after displacement of the movable punch by 87.0 mm.

Fig. 21. Distribution of effective stresses after compression of the billet by the upper movable punch at the velocity 0.5 mm/sec with the height 220.0 mm to the height 133.0 mm.



a. Distribution of effective strain rates after displacement of the movable punch by 40.0 mm.



b. Distribution of effective strain rates after displacement of the movable punch by 87.0 mm.

Fig.22. Distribution of effective strain rates after compression of the billet by the upper movable punch at the velocity 0.5 mm/sec with the height 220.0 mm to the height 133.0 mm

4.1.2. Stress and strain states of the billet out of the titanium alloy Ti-6Al-4V with initial coarse-grained martensite structure subjected to deformation with plastic metal flow in one direction on oblique-angled dies.

The localization of deformation along one shear bands can be removed by changing the angle of inclination of upper and lower portions of the die set. While modeling the angle of inclination of dies were changed. Figures 23, 24, 25, 26 show, respectively, the initial position of the billet and distributions of effective strains, stresses and rates after compression of the billet from 220.0 mm to 133.0 by the movable punch for oblique-angled dies with the angle of inclination 4 degrees. The results show formation of two intersecting bands of deformation localization which significantly increase strain uniformity. In this case no folds are formed.

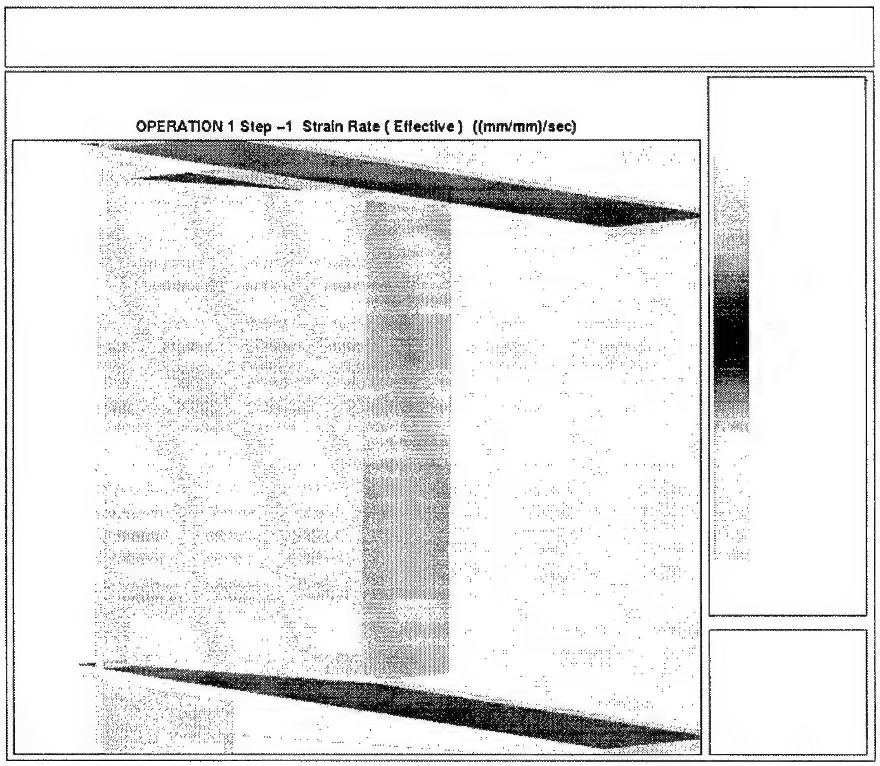
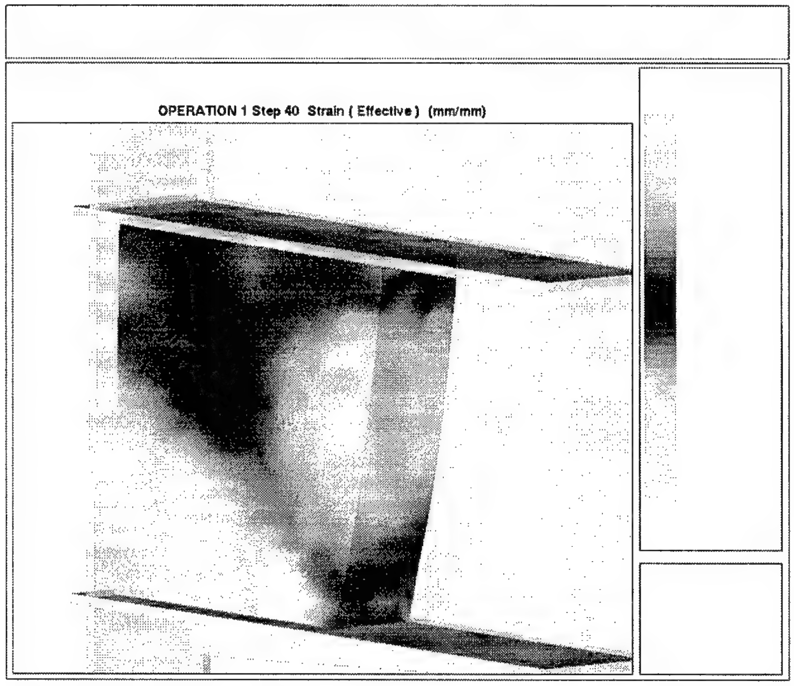
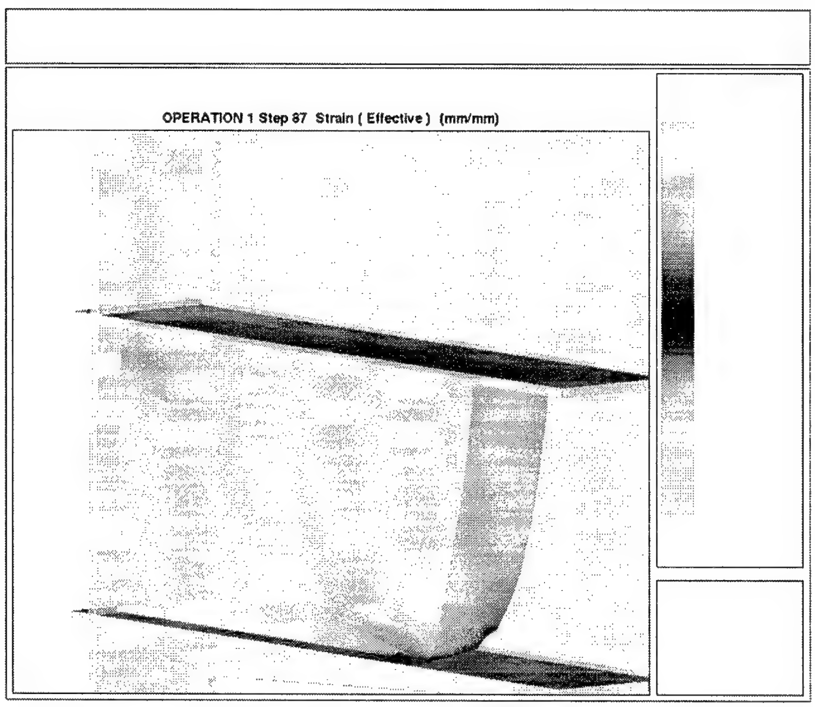


Fig. 23. Initial position of the billet

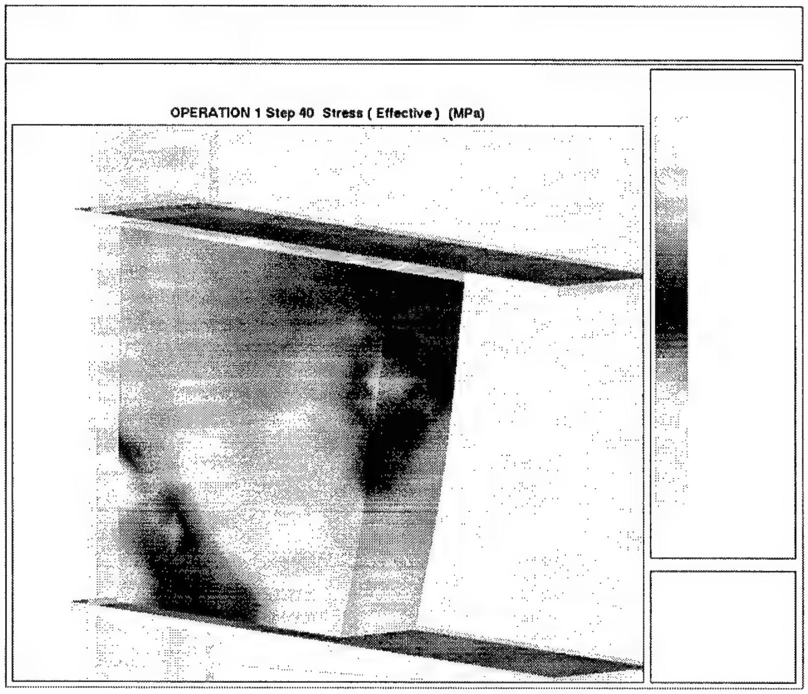


a. Distribution of effective strain after displacement of the movable punch by 40.0 mm

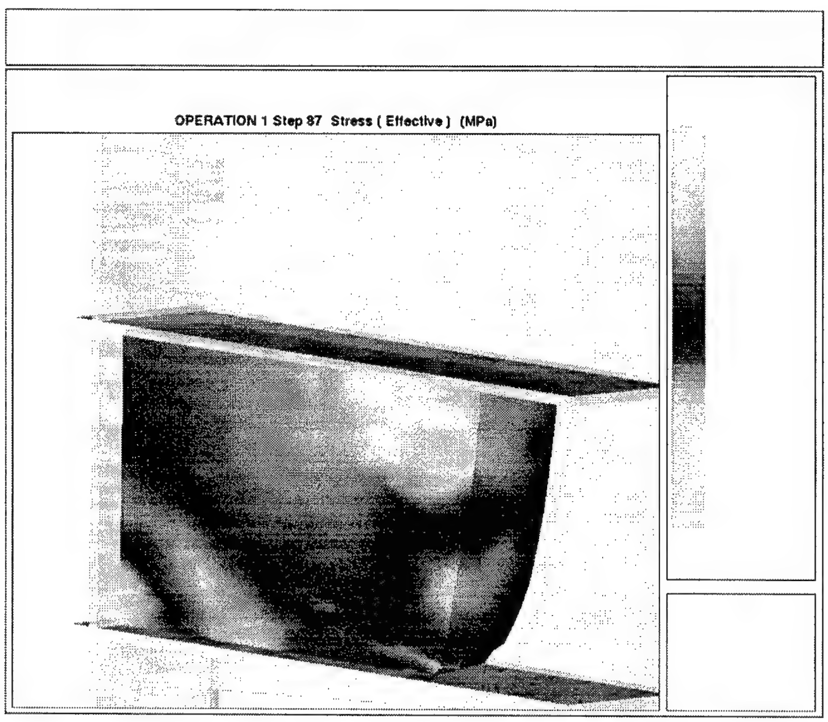


b. Distribution of effective strain after displacement of the movable punch by 87.0 mm

Fig. 24. Distribution of effective strain after compression of the billet by the upper movable punch at the velocity 0.5 mm/sec with the height 220.0 mm to the height 133.0 mm.

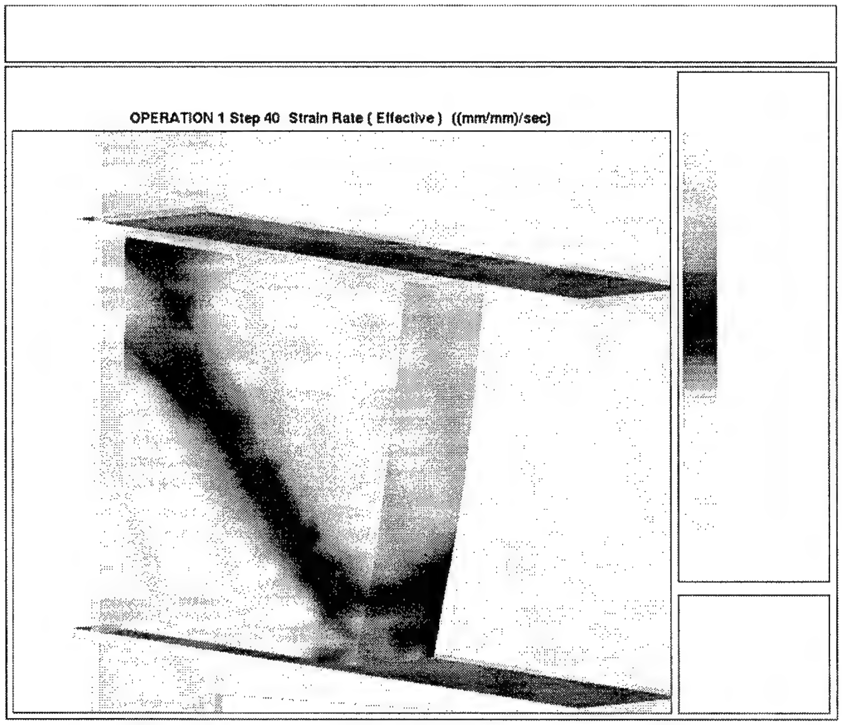


a. Distribution of effective stresses after displacement of the movable punch by 40.0 mm.

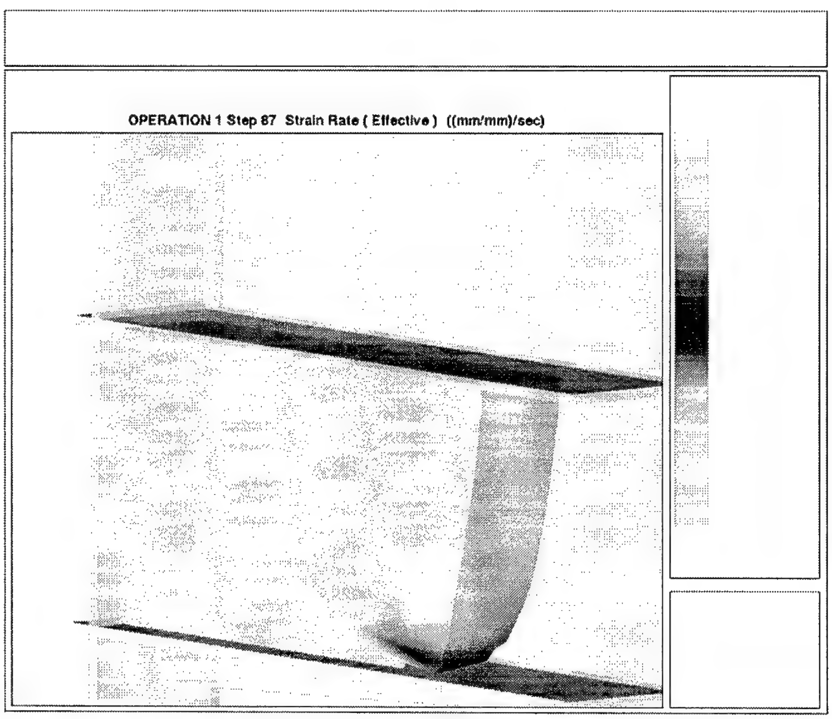


b. Distribution of effective stresses after displacement of the movable punch by 87.0 mm.

Fig. 25. Distribution of effective stresses after compression of the billet by the upper movable punch at the velocity 0.5 mm/sec with the height 220.0 mm to the height 133.0 mm.



a. Distribution of effective strain rates after displacement of the movable punch by 40.0 mm.



b. Distribution of effective strain rates after displacement of the movable punch by 87.0 mm.

Fig. 26. Distribution of effective strain rates after compression of the billet by the upper movable punch at the velocity 0.5 mm/sec with the height 220.0 mm to the height 133.0 mm

4.1.3. Stress and strain states of the billet out of the titanium alloy Ti-6Al-4V with initial coarse-grained martensite structure subjected to deformation with plastic metal flow in two directions.

In this case the billet is installed into the die set in such a way that as the specified high strain achieves 50%, one of its lateral surfaces approaches the vertical bound of the die set. Figures 27, 28, 29, 30 show, respectively, the initial position of the billet and distributions of effective strains, stresses and rates after compression of the billet from 220.0 mm to 133.0. It is seen that in case of such a position of the billet the strain is concentrated mainly in two intersecting bands of deformation localization which significantly increase its uniformity. In this case no folds are formed.

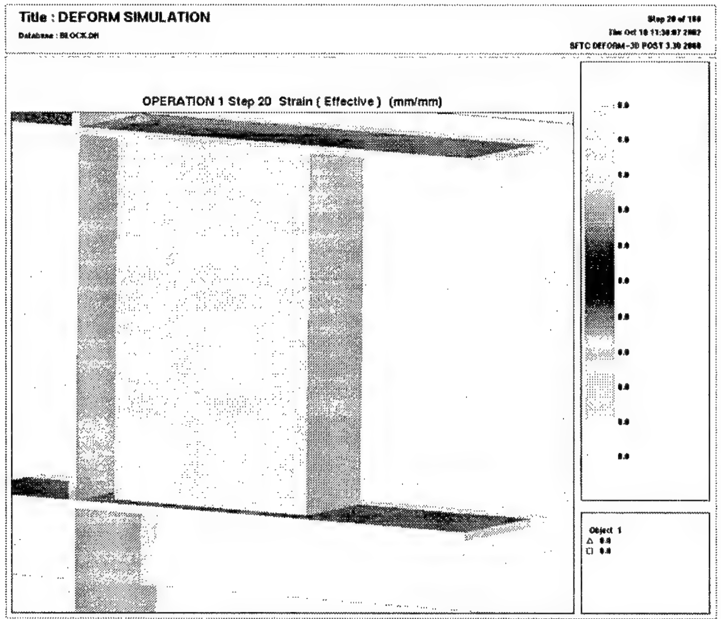


Fig. 27. Initial position of the billet

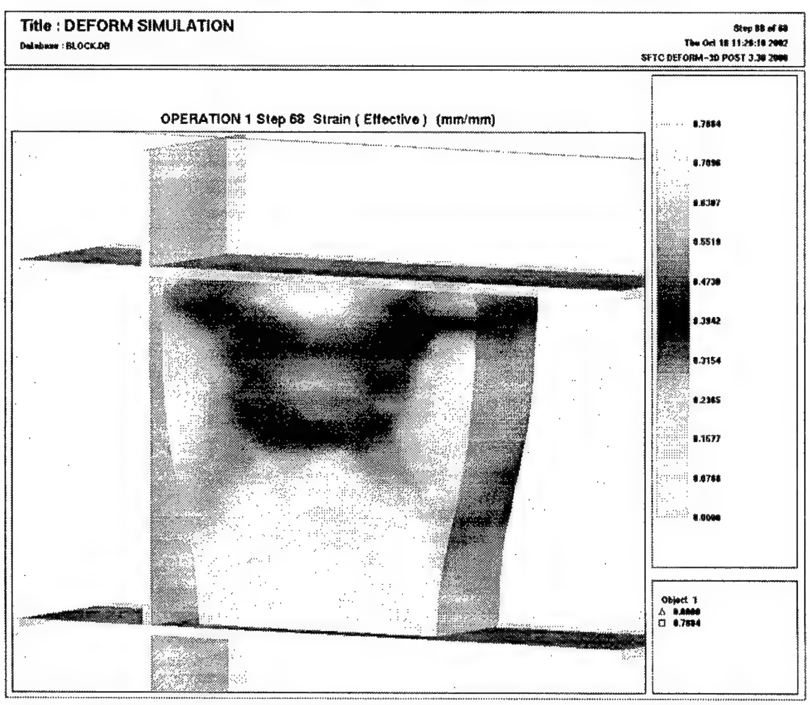


Fig. 28. Distribution of effective strain after compression of the billet by the upper movable punch at the velocity 0.5 mm/sec with the height 220.0 mm to the height 133.0 mm.

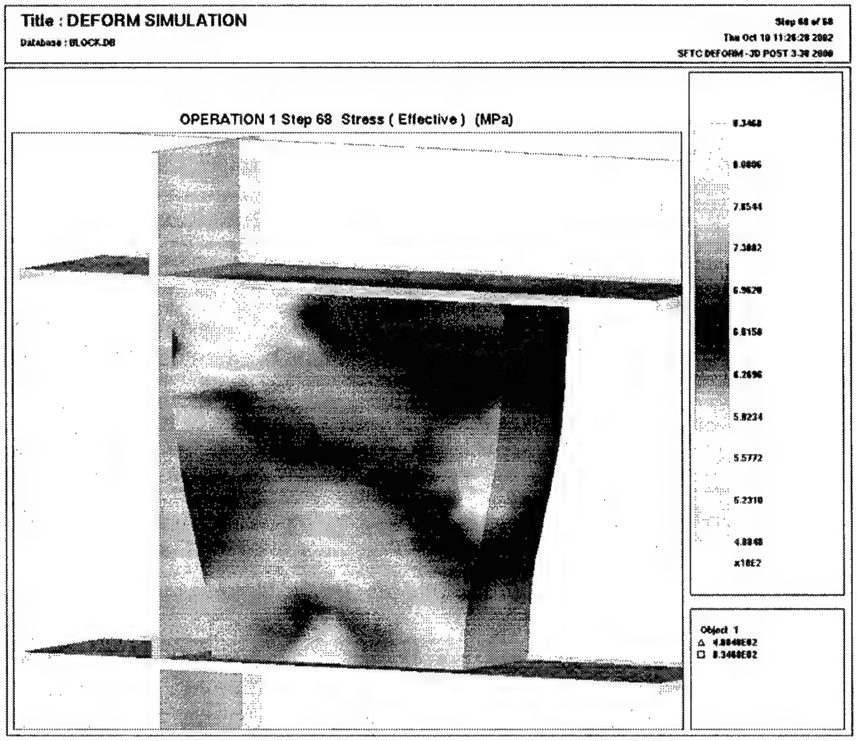


Fig. 29. Distribution of effective stresses after compression of the billet by the upper movable punch at the velocity 0.5 mm/sec with the height 220.0 mm to the height 133.0 mm.

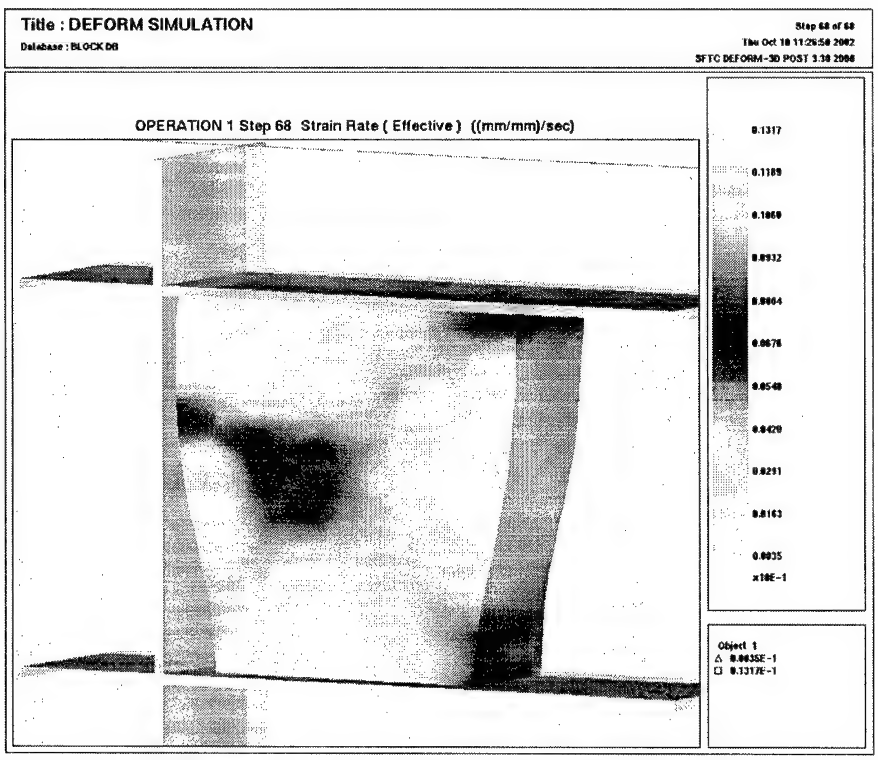
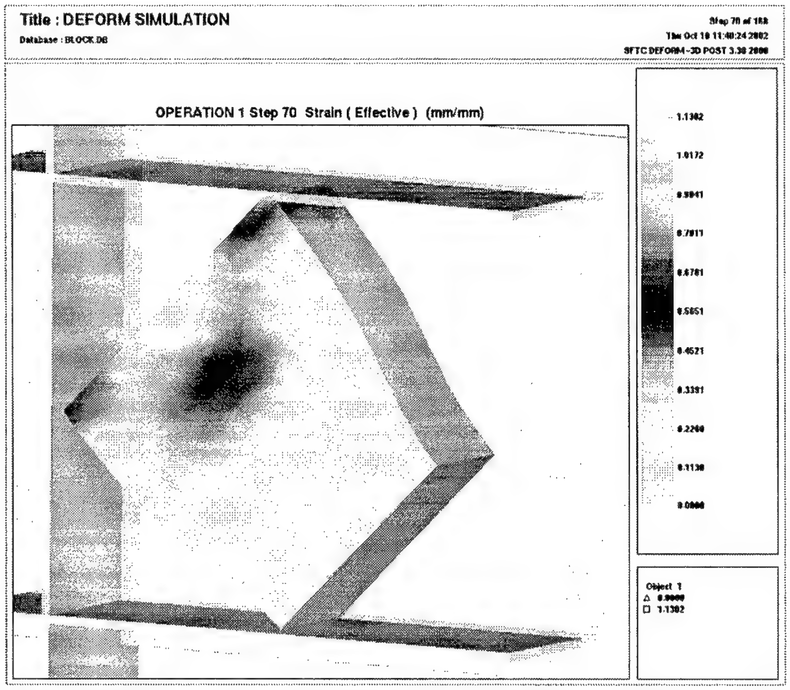


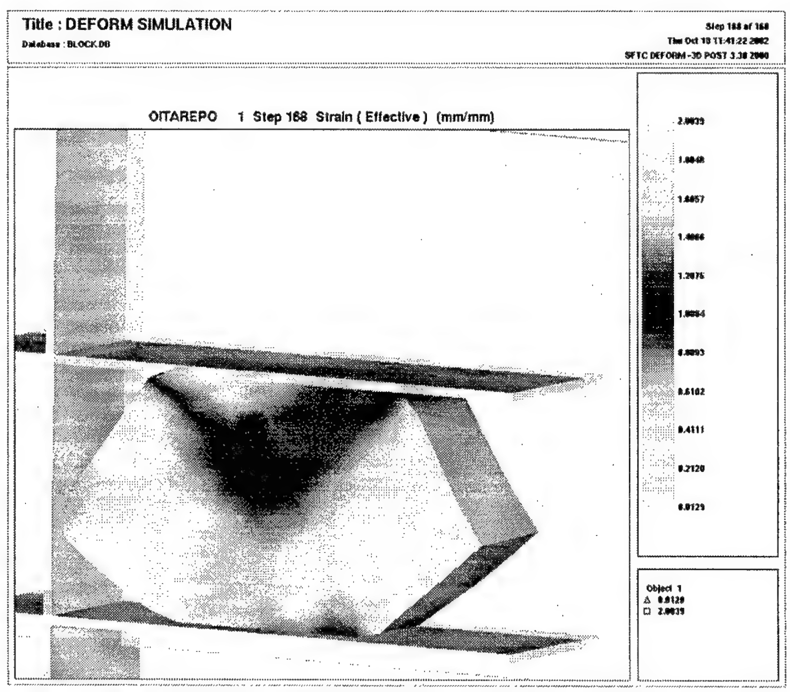
Fig. 30. Distribution of effective strain rates after compression of the billet by the upper movable punch at the velocity 0.5 mm/sec with the height 220.0 mm to the height 133.0 mm

4.1.4. Stress and strain states of the billet out of the titanium alloy Ti-6Al-4V with initial coarse-grained martensite structure subjected to deformation in two stages: 1. as in 1.1.3. with free metal plastic flow in two directions; 2. With a turn of the billet processed over 45 °

After compression of the billet from the height 220 mm to 180 mm, it was turned over the angle 45 degrees. Figures 31, 32, 33, show, respectively, distributions of effective strains, stresses and rates after compression of the billet from 220.0 mm to 133.0 by the upper movable punch. It is seen that in case of such a position of the billet the strain is not concentrated in bands of deformation localization. New bands are formed and they are distributed more uniformly within its interior.

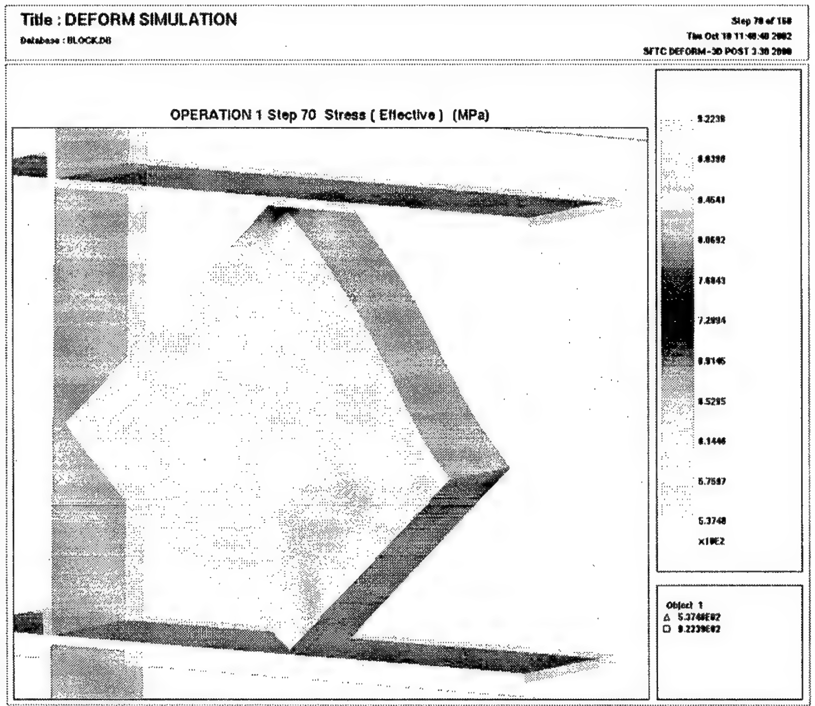


a. Distribution of effective strain after displacement of the movable punch by 10.0 mm

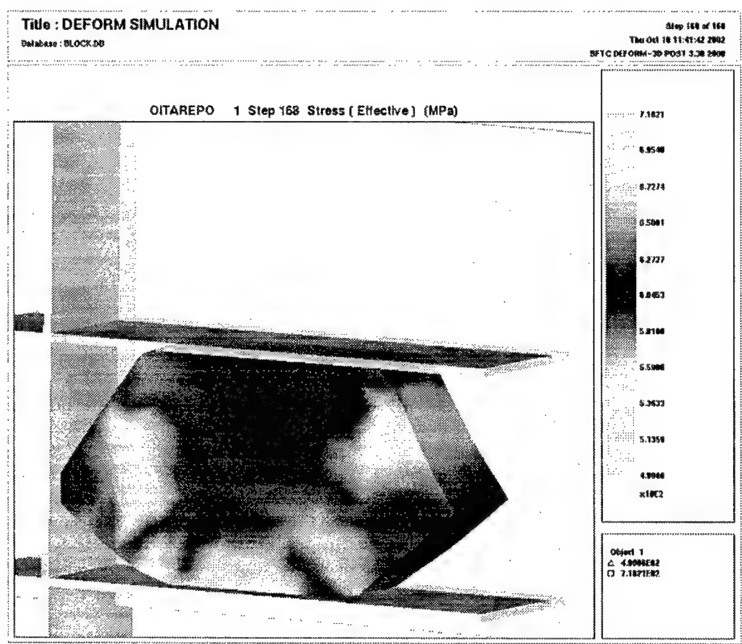


b. Distribution of effective strain after displacement of the movable punch by 107.0 mm

Fig. 31. Distribution of effective strain after turning over the angle 45 degrees and compression of the billet by the upper movable punch at the velocity 0.5 mm/sec with the height 250.0 mm to the height 133.0 mm.

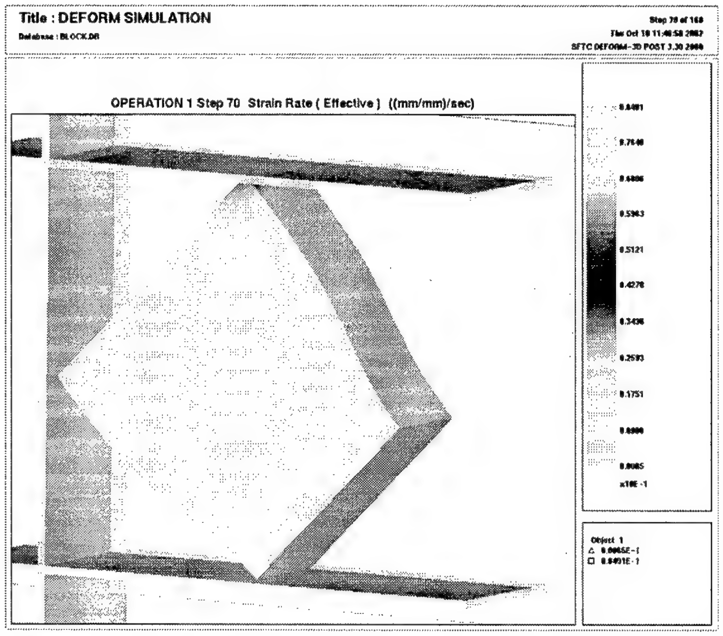


a. Distribution of effective stresses after displacement of the movable punch by 10.0 mm

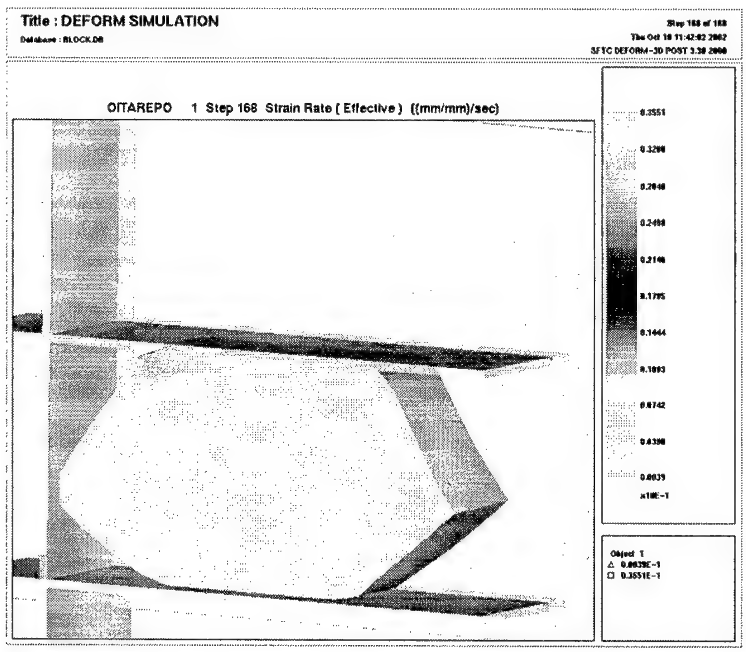


b. Distribution of effective stresses after displacement of the movable punch by 107.0 mm

Fig. 32. Distribution of effective stresses after turning over the angle 45 degrees and compression of the billet by the upper movable punch at the velocity 0.5 mm/sec with the height 250.0 mm to the height 133.0 mm.



a. Distribution of effective strain rates after displacement of the movable punch by 10.0 mm



b. Distribution of effective strain rates after displacement of the movable punch by 107.0 mm

Fig. 33. Distribution of effective strain rates after turning over the angle 45 degrees and compression of the billet by the upper movable punch at the velocity 0.5 mm/sec with the height 250.0 mm to the height 133.0 mm.

4.1.5 Stress and strain states of the billet out of the titanium alloy Ti-6Al-4V with initial SMC structure subjected to deformation with plastic metal flow in one direction.

The results demonstrate final stage of multiple step forging. Figures 34, 35, 36, 37 show respectively, the initial position of the billet and distributions of effective strains, stresses and rates after compression of the billet by the upper movable punch from 220.0 mm to 133.0. The results of modeling demonstrate essentially more uniform distribution of evaluated parameters, than in the billet with initially coarse grained martensite structure. However, possibility of lap formation at such disposition of the sample required the control under shape of the billet. This problem can be solved by using oblique-angled dies (Part 4.1.2) or changing the placement of the billet (Part 4.1.3). Using the results of modeling the die tooling allowing to increase workability of the material and produce large scale billets with SMC structure has been designed and manufactured.

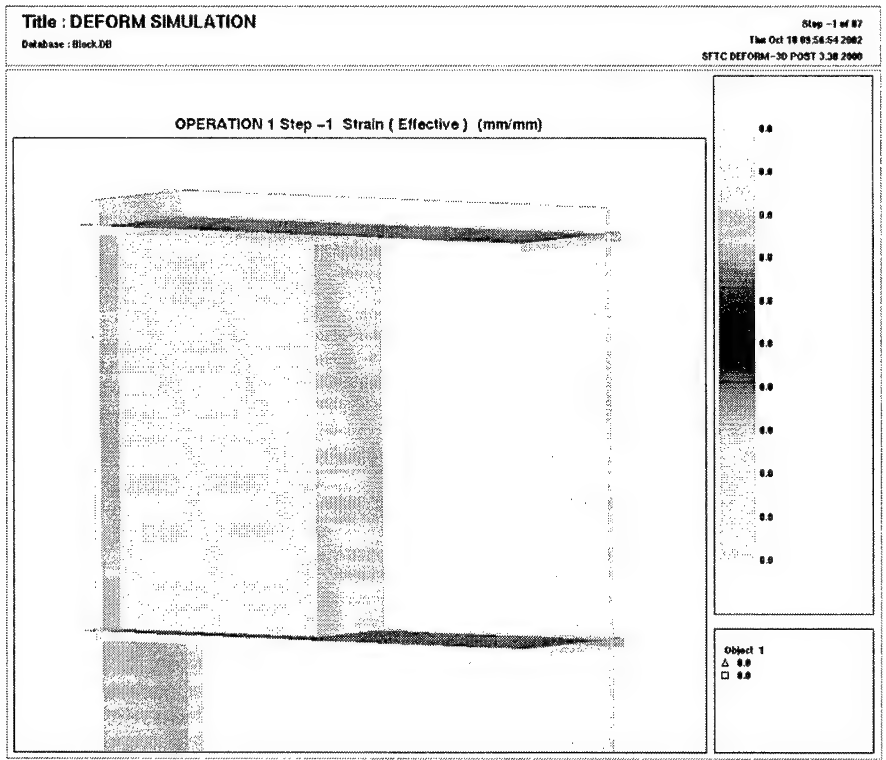
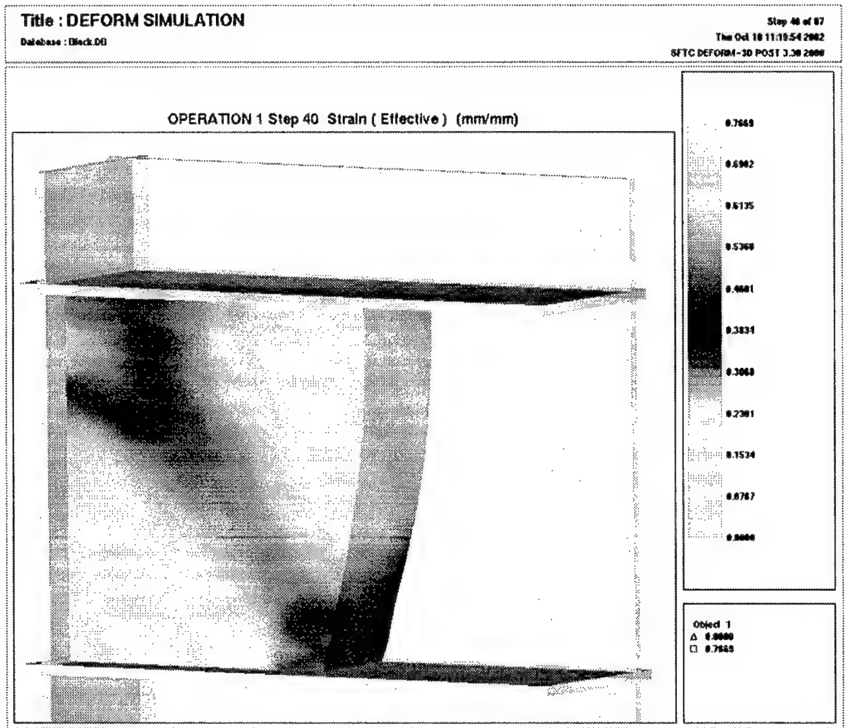
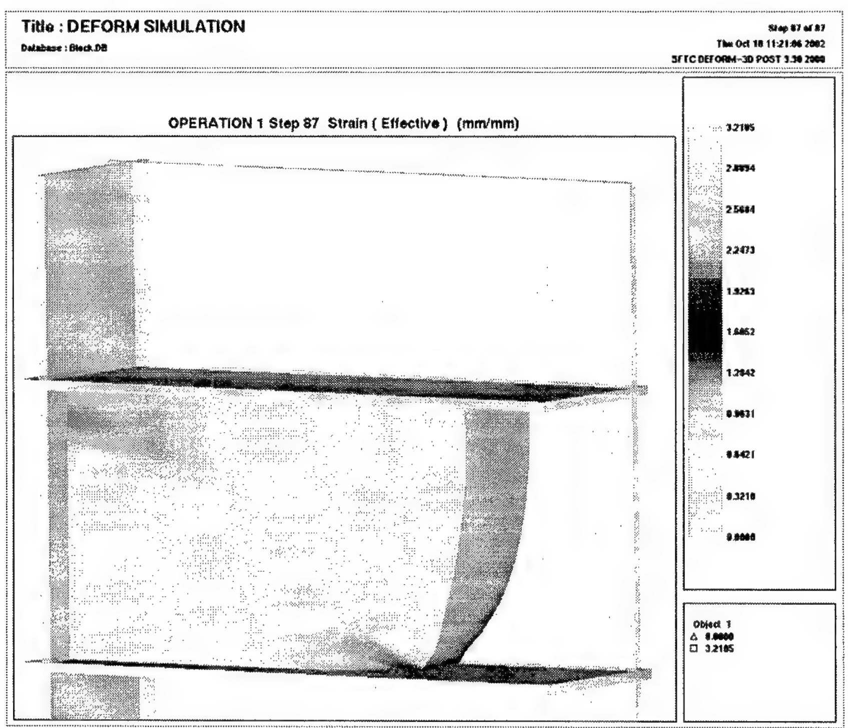


Fig. 34. Initial position of the billet

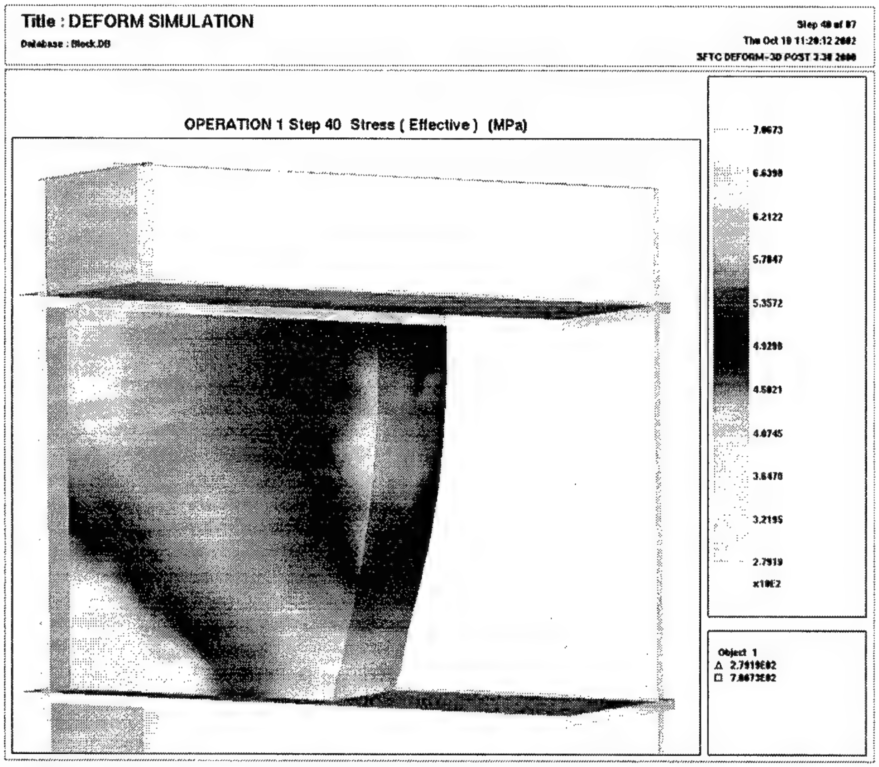


a. Distribution of effective strain after displacement of the movable punch by 40.0 mm

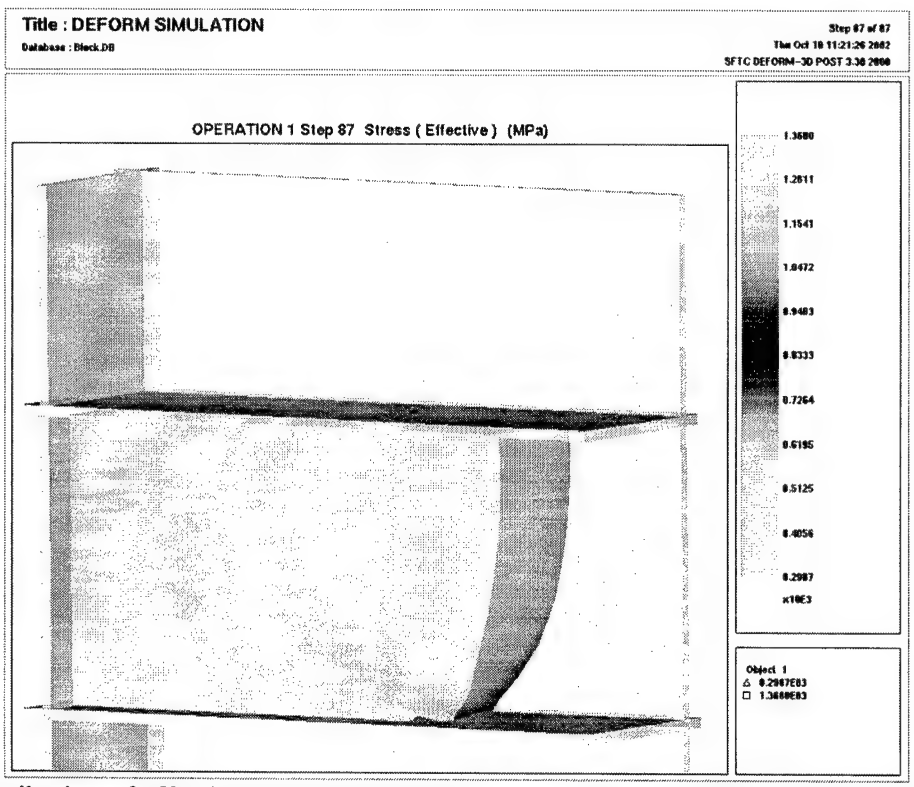


b. Distribution of effective strain after displacement of the movable punch by 87.0 mm

Fig. 35. Distribution of effective strain after turning over the angle 45 degrees and compression of the billet by the upper movable punch at the velocity 0.5 mm/sec with the height 220.0 mm to the height 133.0 mm.

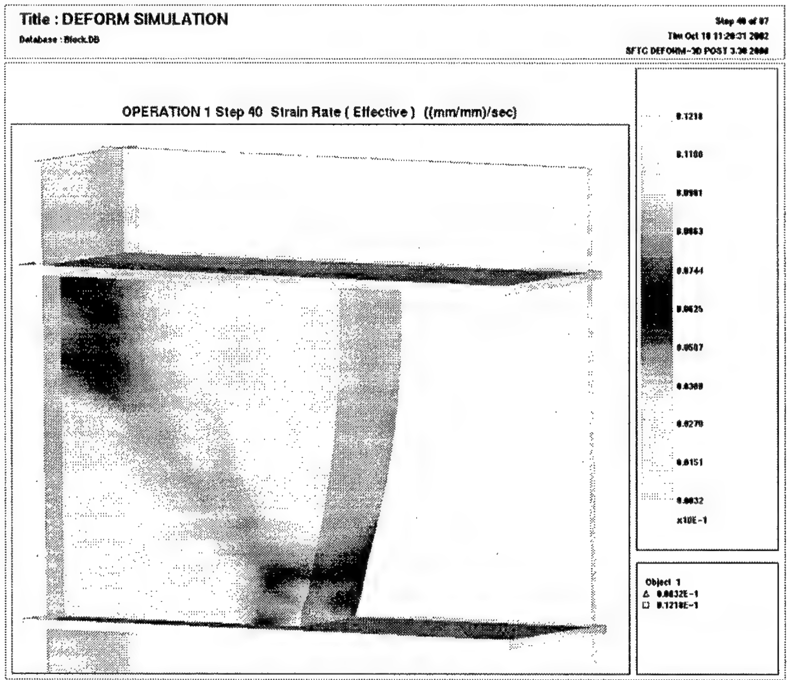


a. Distribution of effective stresses after displacement of the movable punch by 40.0 mm

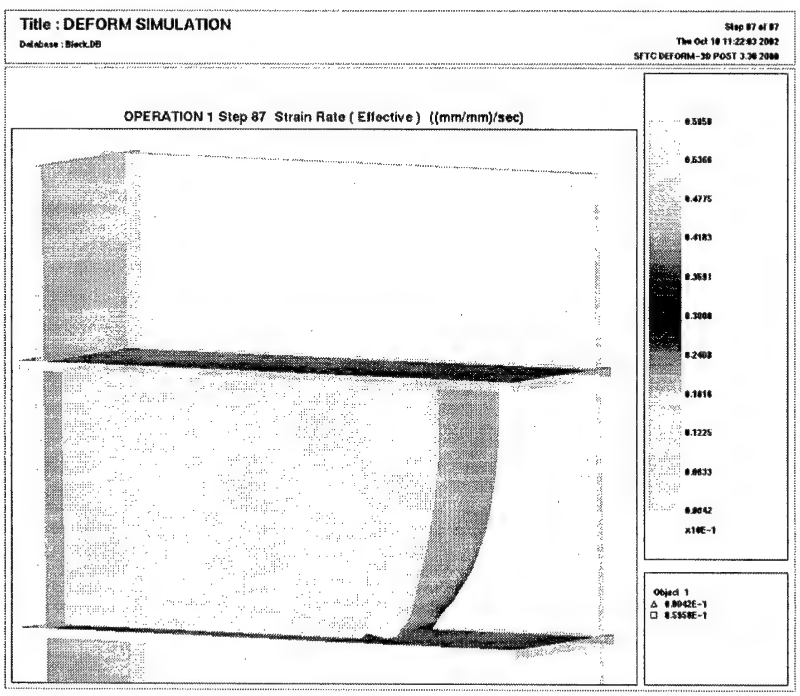


b. Distribution of effective stresses after displacement of the movable punch by 87.0 mm

Fig. 36. Distribution of effective stresses after turning over the angle 45 degrees and compression of the billet by the upper movable punch at the velocity 0.5 mm/sec with the height 220.0 mm to the height 133.0 mm.



a. Distribution of effective strain rates after displacement of the movable punch by 40.0 mm



b. Distribution of effective strain rates after displacement of the movable punch by 87.0 mm

Fig. 37. Distribution of effective strain after turning over the angle 45 degrees and compression of the billet by the upper movable punch at the velocity 0.5 mm/sec with the height 220.0 mm to the height 133.0 mm.

A-4.2. Manufacture of die tooling for producing billets, ∅150×200 mm, with homogeneous microstructure and a grain size less than 0.5 μm.

The set of technical documentation on design of special tooling for 'abc' isothermal forging of massive blanks with uniform microstructure has been developed.

The Agreement between ISTC and OJSC "INITCIATIVE PLUS on delivery of large size ingots out of high temperature alloys for manufacturing this die tooling has been executed.

The ingots out of high temperature nickel base alloys delivered by OJSC "INITCIATIVE PLUS" pursuant to Agreement 1-01 for manufacturing tooling have undergone machining.

Ingots out of the high-temperature nickel base alloy ZhS6 have been manufactured according to the design drawings and special die tolling was produced (Figs. 38. 39). The induction heater to the die tooling has been fabricated.

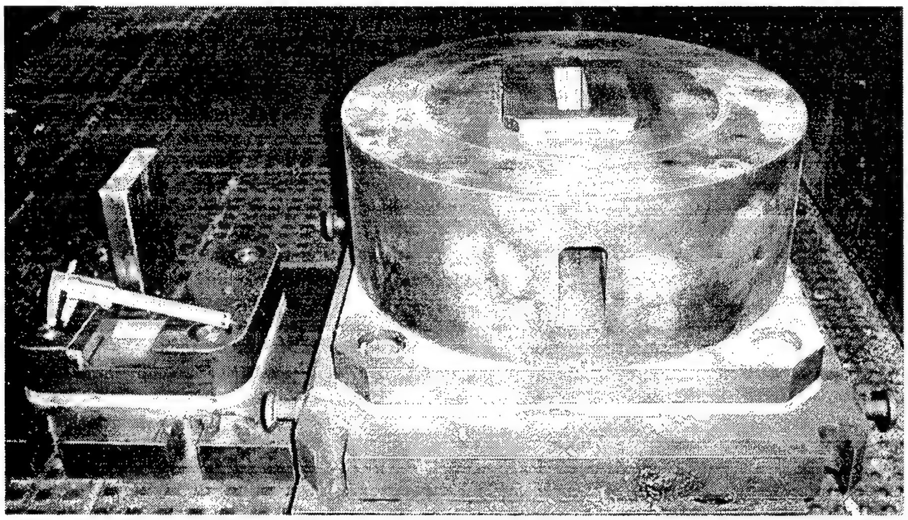


Fig. 38. Special die tooling

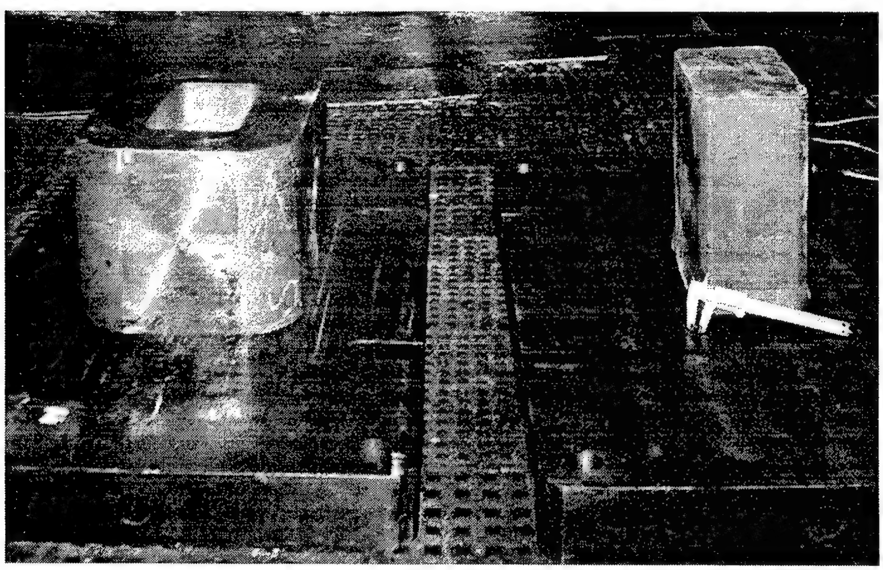


Fig. 39. Special die tooling for fabrication large scale billets

Activity-5. Development of the pilot technology for producing massive billets, ∅150×200 mm, with a grain size less than 0.5 µm.

On the basis of the investigations performed under the Project one can determine a number of requirements on selection of temperature-strain rate regimes of deformation and initial microstructure of billets which are very important for developing a pilot technology for producing large scale billets with SMC structure by multiple step isothermal forging. These requirements are the following:

1. In the alloy Ti-6Al-4V the grain size less than 0,5 µm is formed at the temperature of deformation not exceeding 600°C and the strain rate $10^{-3}sec^{-1}$.

2. Since critical strains until initiation of first features of globularization increase sharply with decreasing temperature, it is necessary that for formation of SMC structure within the whole sample interior the strain values should be no less than Σe=3.

3. For formation of SMC structure it is more preferable to use the alloy with the initial martensite structure or perform intermediate processing of the alloy for formation of globular type microstructure in which areas of β-transformed constituents are absent.

4. For conducting deformation at 550°C and the initial martensite structure of the billet it is necessary that the allowable strains until initiation of cracks on its lateral surface should not exceed 55% and these strains are reached at high deformation no more than 50%.

A-5.1. Investigation of an evolution of initial microstructure of the rod in the as-received and processed states.

The forged rod, 230 mm in diameter, out of alloy Ti-6Al-4V purchased by Verkhnaya-Salda Metallurgical Production Association was used as material for producing a billet, ∅150×200 mm. . The chemical composition of the alloy under study is shown in Table 1. The temperature of polymorphic transformation of the rod material was 995°C. The microstructure of the alloy in the as-received condition is shown in Fig. 1. Billets with the dimensions ∅ 230×100mm were cut out from the rod for following processing. The billet was subjected to heat treatment: heating in β-region up to 1010°C/1 h., cooling in water. The mean β-grain size did not change as compared to the initial material and was equal to 1000 µm.

A A-5.2.Development of a technological route for processing billets, ∅150×200 mm, with uniform microstructure and a grain size less than 0.5 µm.

On the basis of the results of investigations the multiple step forging of the billet has been performed. The technological route for processing the billet was the following :

- temperature of forging was 600 ^{0}C
- strain value at each pass of forging was no more than 40%
- strain rate was within the range 5×10^{-3}-10^{-3} sec^{-1}
- after reaching the specified strain value the billet is turned at the pass as shown in Fig.8.
- the total strain was no less than Σe=3.

Activity A-6. "Production of large size billet with dimensions ∅150×200mm with uniform SMC structure (d ≤ 0.5 µm) for joint study".

A-6.1. Production of a billet with dimensions ∅150×200mm with uniform microstructure with grain size not exceeding 0.5 µm.

On the basis of the technological route developed the massive billet 150 in diameter and 200 mm in length with homogenous structure was produced (Fig.40).

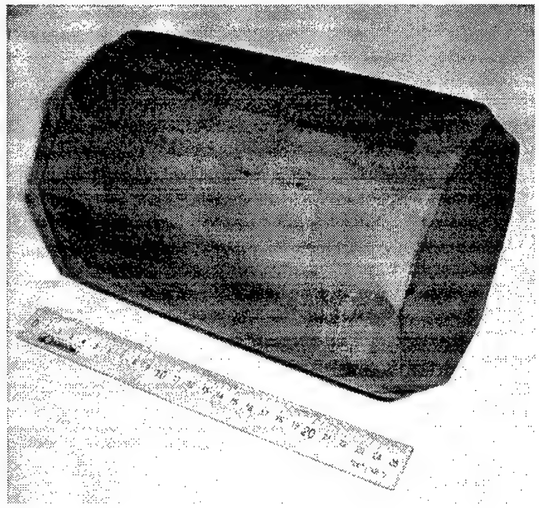


Fig. 40. SMC billet out of the alloy Ti-6Al-4V.

A-6.2. Evaluation of quality and properties of the billet processed.

To evaluate the microstructure and mechanical properties of the billet produced a templet, about 20 mm in thick, was cut out from the billet face. A macrosection was made on the billet surface formed. Standard samples were cut out from the templet in radial and tangential directions with the gauge diameter 5 mm for microstructure studies.

The macrosection of the billet face is shown in Fig. 41. It is seen that the billet has a homogeneous macrostructure along the whole section and separate grains are not revealed on the macrosection.

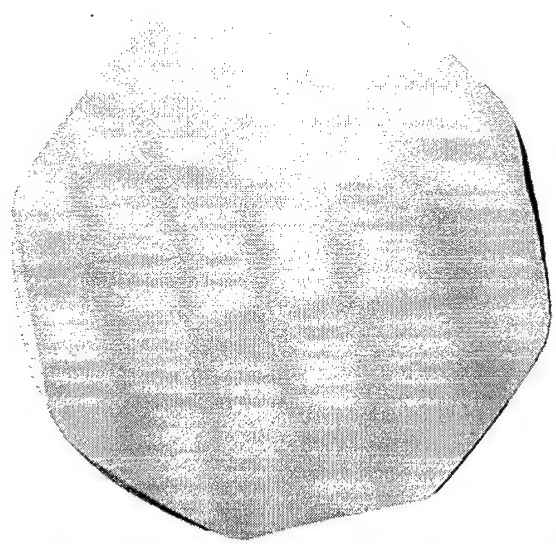


Fig. 41. Macrostructure of uniform SMC processed billet
(150 mm in diameter and 200 mm in length) out of Ti-6Al-4V alloy.

The microstructure of the billet is characterized by uniformly distributed globular particles of α-and β- phases, which size does not exceed 0.5 μm (Fig. 42a,b).

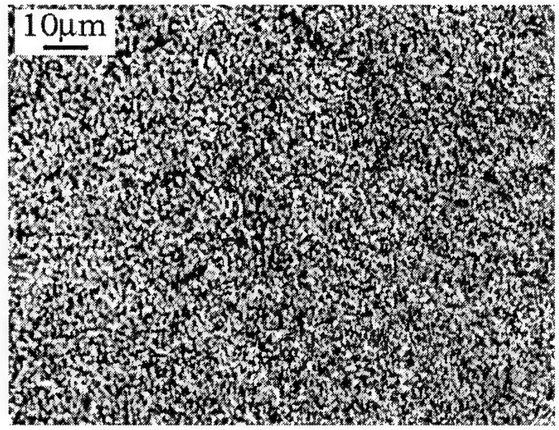


Fig. 42. Fine structure (a) and microstructure (b) of uniform SMC processed billet (150 mm in diameter and 200 mm in length) out of Ti-6Al-4V alloy.

Mechanical properties of samples in radial and tangential directions are shown in Table 4.1.3. It is seen that after the treatment proposed a value of ultimate tensile strength of the processed alloy is rather high and equal to 1360 MPa and its elongation is 7%. Whereas, after conventional heat treatment including solution treatment and aging the alloy has ultimate tensile strength no higher than 1100 MPa and ductility of about 8%. One should note high uniformity of mechanical properties in different directions that testifies the absence of anisotropy of properties in the billet.

Table 4.1.3

Mechanical properties of samples cut out from uniform SMC processed billet out of Ti-6Al-4V alloy.

Direction	YS, MPa	UTS, MPa	EL,%	RA,%
Radial	1350	1360	7	62
Tangential	1335	1355	7	61

5. Conclusion

All Project activities have been fulfilled in full scope. The following main results have been attained:

1. The influence of deformation temperature on formation of globularized microstructure was studied in the Ti-6Al-4V titanium alloy with initial coarse-grained martensite type structure It has been shown that the decrease in deformation temperature decreases the size of forming globularized grains and increases critical strains of their initiation. The grains size less than 0,5 μm is formed at the deformation temperature exceeding 600°C and the strain rate $10^{-3}sec^{-1}$. It has been established that the relationship between the steady flow stress and the globularized grain size can be described by the equation $\sigma_s = kd^{-n}$, and experimental data are well approximated by the straight line with the exponent n=1.
2. Mechanical behavior and evolution of microstructure of the Ti-6Al-4V alloy with initial coarse-grained martensite type microstructure were studied in the process of successive compressive straining of samples in three orthogonal directions at 800 and 550°C and the strain rate $10^{-3}s^{-1}$. It has been shown that true flow stress-cumulative deformation (S-Σe) curves for both temperatures are similar. They have a peak of flow stress, and stages of softening and steady flow The value of

the strain rate sensitivity coefficient m and the value of activation energy in the stage of steady flow at the temperature 550°C are equal to 0,35 and 186 kJ/mol, respectively, and at 800°C - 0,42 and 210 kJ/mol. Values m>0,3 and values Q are close to the known values for superplastic flow of this alloy and testify the development of superplasticity in the final stage of transformation of the coarse-grained lamellar microstructure to the globular one. The results obtained demonstrate that it is possible to use successive deformation in three orthogonal directions for formation of homogeneous SMC structure in billets out of the Ti-6Al-4V titanium alloy. The cumulative strain of samples should be no less than $\Sigma e=3$.

3. It has been studied the influence of the initial microstructure type of the alloy Ti-6Al-4V: martensite (water cooling from β-region), lamellar (air cooling from β-region), globular (multiple step isothermal forging at 700°C) and "bi-modal" (primary α in transformed β) (multiple step isothermal forging at 950°C with following air cooling), on formation of homogeneous SMC structure during deformation at the temperature 550°C. It has been established that for formation of homogeneous SMC structure it is more preferable to use the alloy in the condition with the initial martensite structure or perform intermediate processing of the alloy for formation of globular type microstructure where areas of β-transformed constituents are absent.
4. It has been studied the influence of the initial microstructure of the alloy Ti-6Al-4V on its workability at the temperature 550°C and the strain rate $10^{-3}sec^{-1}$. It has been shown that the alloy with initial martensite type structure displays higher ductility and larger strains until formation of cracks than the alloy with coarse lamellar structure. The alloy with a globular type microstructure demonstrates the highest workability.
5. Performance of mathematical 3D modeling using DEFORM-3D for determination of the optimal route of the technological process and the geometry of the special die tooling to produce massive billets out of the Ti-6Al-4V alloy with homogeneous microstructure and a grain size less than 0.3 μm. The results obtained provide determining the most optimal die set geometry, the installation of the billet in the die set and the sequence of strain operations.
6. The results of studies of the influence of temperature-strain rate conditions of deformation and initial microstructure on mechanical behavior, evolution of microstructure and workability of the Ti-6Al-4V alloy and their analysis allowed developing the method of multiple step isothermal forming for processing large scale billets with SMC structure.
7. Using the method developed two large scale billets, 150 mm in diameter and 200 mm in length, with homogeneous microstructure were produced. Analysis of their quality has shown that the homogeneous microstructure with a grain size less than 0.5 μm was formed in them. Investigations of mechanical properties have shown that strength and ductile characteristics of the billets produced are close in radial and tangential directions.

6. References

1. R.Z. Valiev, R.K. Islamgaliev, I.V. Alexandrov, Bulk Nanostructured Materials from Severe Plastic Deformation, Progr. Mater. Sci., 45 (№2), (2000), p. 102-189
2. A. Belyakov, H. Miura, T. Sakai, Mat. Sci. Eng., 1998, A255, pp. 139-147.
3. Rh. Bocher and J.J. Jonas in Recrystallization and Relation Phenomena (ReX'99')", eds. T. Sakai and T. Tsuzaki, Japan Inst. Metals, 1999, pp. 25-35.
4. A. Belyakov, T. Sakai, H. Miura, Fine-grained Structure Evolution in Austenitic Stainless Steel under Multiple Warm Deformation at 0,5 T_m, Mat. Trans., JIM, 2000, v. 41, №4, pp. 476-484.
5. S.V.Zherebtsov, G.A.Salishchev, R.M. Galeyev. Dynamic Recrystallization of Titanium and Ti-6Al-4V Two-Phase Titanium Alloy, Recrystallization and Grain Growth: Proceeedings of the First Joint International Conference, August 37-31, 2001, RWTH Aachen, Germany, vol.II, pp. 961-966.
6. N. Hansen, X. Huang, D.A. Hughes. High Monotonic Deformation-Structure and Strength. Proceedings of "Ultrafine Grained Materials II", Y.T. Zhu, T.G. Langton, R.S. Mishra, S.L.

Semiatin, M.J. Saran, and T.C. Lowe, (eds), TMS Annual Meeting in Seattle, Washington, February 17-21, 2002, pp. 3-14.

7. G.A.Salishchev, S.V.Zherebtsov, R.M. Galeyev, Evolution of Microstructure and Mechanical Behavior of Titanium During Warm Multiple Forging. // Proceedings of "Ultrafine Grained Materials II", Y.T. Zhu, T.G. Langton, R.S. Mishra, S.L. Semiatin, M.J. Saran, and T.C. Lowe, (eds), TMS Annual Meeting in Seattle, Washington, February 17-21, 2002, pp. 123-131.
8. G.A. Salischev, S.V. Zherebtsov, S.Yu. Mironov, Formation of Mesoscopic Scale Structure and Mechanical Behaviour of Titanium at the Large Plastic Deformation, submitted to the «Questions of Material Science» (in Russian).
9. Humphreys F.J., Hatherly M. Recrystallization and Related Annealing Phenomena. Pergamon, 1996, p.497.
10. A.W. Thompson, Met. Trans., 1977, 8A, pp.833-842.
11. T. Sakai, A. Belyakov, H. Miura, Dynamic Recrystallization under Continuous and Discontinuous Reation. Recrystallization and Grain Growth: Proceeedings of the First Joint International Conference, August 37-31, 2001, RWTH Aachen, Germany, vol.II, pp. 669-682.
12. O.A. Kaibyshev, R.Ya. Lutfullin, G.A. Salishchev " An Influence of Superplastic Deformation Conditions on the Lamellae Microstructure Transformation in VT9 titanium alloy" in Fiz. Met. Metalloved. 1988, v.66, № 6, pp.1163-1171 (in Russian).
13. O. A. Kaibyshev. Sverkhplastichnost' Promyshlennykh Splavov (Metallurgia, Moscow 1984) p 184.
14. G.A. Salishchev, O.R. Valiakhmetov, R.M. Galeev, Formation of Submicrocrystalline Structure in the Titanium Alloy VT8 and its Influence on Mechanical Properties, J.Mater.Sci., 1993, v.28, p.2898-2902.
15. M.A. Murzinova, G.A. Salishchev, D.D.Afonichev. Formation of Submicrocristalline Structure in Titanium Alloys by Combination of Termohydrogen Processing with Hot Working, Intern. J. Hydrogen Energy.- 2002, Vol. 27, No. 7-8, p. 775-782.
16. A.A. Baranov O nachalnih stadiyah sferoidizacii cementita v stalyah, Izv. AN SSSR. Mettali, 1969, №3, c. 104-107 (in Russian).
17. M. McLean, Microstructural Instabilities in Metallurgical System, Met. Sci. Rev. Paper., 1978, V.3, p.113-121.
18. H. Pacueton, A. Pineau, Acseleration of Pearlite Spheroidization by Thermomechanical Treatment, Iron and Steel Institute, 1971, V209, N2, p.991-998.
19. Mazurski M.I., Salishchev G.A. Effect of Interface Anisotropy on the Thermal Stability and Transformation of Lamellar Structures, Phys. Stat. Sol. (b) 1995, v.188, p.653-658.
20. V.K. Alexandrov, N.F. Anoshkin, G.A. Bochvar et al., "Semi-manufactured articles made of titanium alloys". ("Metallurgy" Publishing House, Moscow, 1979) (in Russian).
21. I.I. Novikov, V.K. Portnoy Optimization of Heterogeneity as General Principle of Controlling Alloys Structure for Superplastic Forming, in Superplasticity and Superplastic Forming, ed. by C.H. Hamilton and N.E. Paton, The Minerals, Metals and Materials Society, 1988, pp. 401-406.
22. G.V. Shakhanova, I.V. Rodina, F.V. Tulyankin, Resistance to Deformation and Dynamic Changes in the Structure of Two-Phase Titanium Alloys under Hot Deformation, Proc. Fourth Intern. conf. on Titanium, Kyoto, Japan, 1980, pp. 849-861.
23. J. Weiss, F.H. Froes, D. Eylon, G.E. Welsch. Modification of Alpha Morfology in Ti-6Al-4V by Thermomechanical Processing. Met. Trans., 1986, V.17A, p.1935-1947.
24. Semiatin S.L., Thomas J.F., Jr. and Dadras P. Processing-Microstructure Relationship for Ti-6Al-2Sn-4Zr-2Mo-0.1Si, Metall Trans. A, 1983, 14A, pp.2363-2374.
25. H. Margolin, P. Cohen, Evolution of the Equiaxed Morphology of Phases, Ed. by H. Kimura, O. Izumi, Titanium'80: Science and Technology, Warrendale, PA, The Minerals, Metals and Materials Society, 1980, pp.1555-1561.
26. H. Margolin, P. Cohen, Kinetics of recrystallization in alpha in Ti-6Al-4V, Ed. by H. Kimura, O. Izumi, Titanium'80: Science and Technology, Warrendale, PA, The Minerals, Metals and Materials Society, 1980, pp.2991-2997.
27. G.A. Salishchev, R.Ya. Lutfullin "Dynamic Recrystallization of Titanium Alloy Ti-5Al-2,5Sn" In

Superplasticity and Superplastic Forming. Ed. by C.H. Hamilton and N.E. Paton, The Minerals, Metals and Materials Society, 1988, pp. 183-186.

28. G.A. Salishchev, O.A. Kaibyshev, M.I. Gordienko, Vliyanie ishodnoi structuri na effect sverhplastichnosti titanovogo splava BT3-1. Izv. Vuzov, Tscvetnaya metallurguya, 1976, №4, c. 104-107.
29. K.K. Chen, Influence of Initial Microstructure on ductility of Ti-6Al-4V alloy and Microstructure after Isothermal Forging, Proc. Third Intern. Conf. on Titanium, Moscow, Russia, 1977, pp. 239-246.
30. H. Gegel, S. Nadiv, a.o. Dynamic Effect on Flow and Fracture During Isothermal Forging of a Titanium Alloy, Scr. Met., 1980, V.14, N2, p.241-246.
31. G.A. Salishchev, M.I. Mazursky, I. E. Levin, An Influence of Phase Nonequilibrium on the Lamellar Microstructure Globularization in VT22 Titanium Alloy during Hot Deformation, Fiz. Met. Metalloved. 1990, v.12, pp.149-151 (in Russian).
32. Y.-S. Him, J. Kim, I. Kim, D.H. Shin, Grain Refifning Mechanism of Ti during Equal Channel Angular Pressing, In Proceedings of "Ultrafine Grained Materials II", Y.T. Zhu, T.G. Langton, R.S. Mishra, S.L. Semiatin, M.J. Saran, and T.C. Lowe, (eds) TMS Annual Meeting in Seattle, Washington, February 17-21, 2002, pp. 99-104.
33. V.V. Stolyarov, Y.T. Zhu, T.C. Lowe, R.K. Islamgaliev and R.Z. Valiev " A Two Step SPD Processing of Ultrafine-Grained Titanium" In NanoStruct. Mat., 1999, v. 11, No 7, pp. 947-954.
34. D.P. DeLo, S.L. Semiatin, Deformation of Ti-6Al-4V via Equal Channel Angular Extrusion, Proceedings of "Ultrafine Grained Materials II", Y.T. Zhu, T.G. Langton, R.S. Mishra, S.L. Semiatin, M.J. Saran, and T.C. Lowe, (eds)TMS Annual Meeting in Seattle, Washington, February 17-21, 2002, pp. 539-546.
35. G.A. Salishchev, Superplasticity of Two-Phase Titanium Alloys, PhD Thesis, MATI, Moscow, 1978.
36. O.A. Kaibyshev, G.A. Salishchev, E.I. Razuvaev, A.F. Matveenko, S.G. Silina, The Method of $\alpha+\beta$ Titanium Alloy Processing, Russian Patent N 676044, 1976.
37. S.L. Semiatin, V Seetharaman and I Weiss "Hot Working of Titanium Alloys – an Overview" In Advances in the Science and Technology of Titanium Alloy Processing, Ed. by I Weiss, R. Srinivasan, P.J. Bania, D. Eylon and S.L. Semiatin, The Minerals, Metals and Materials Society, 1997, pp. 3-73.
38. A.A. Ilyin Mehanizm I kinetika fazovih i structurnih prevrachenii v titanovih splavah. – M.: Nauka, 1994, 304c.
39. M.L. Meier, D.R. Lesuer, A.K. Mukherjee, α Grain Size and β Volume Fraction Aspects of the Superplasticity of Ti-6Al-4V, Mater. Sci. and Eng. 1991, A136, pp. 71-78.
40. RF Patent "A Method for Processing Titanium Alloy ", by, Kaibyshev O.A., Salishchev G.A., Galeyev R.M., Lutfullin R.Ya. and Valiakhmetov O.R., number is №96120958/02(027459) 1996.
41. T. Matsumoto, M. Nishigaki, M. Fukuda and T. Nishimura, Effect of Heat Treatment on the Surface Defect and Mechanical Properties of Ti-6Al-4V Rolled bar, Ed. by G. Luetjering, U. Zwicker, and W. Bunk, Titanium: Science and Technology, Oberursel, Germany: Deutsche Gesellschaft fur Metallkunde e.V. 1985, pp.617-683.
42. S.L. Semiatin, V. Seetharaman, unpublished research, Wright Laboratory Materials Directorate, Wright-Patterson, AFB, OH, 1995.
43. H.G. Suzuki, D. Eylon, Hot Ductility of Titanium Alloys – A Comparison with Carbon Steels, ISIJ Inter., 1993, 33, pp.1270-1274.
44. RF Patent "A method for processing billets and die tool for its performance (Versions)", by Valiakhmetov O.R., Galeyev R.M., Kaibyshev O.A., and Salishchev G.A., its priority number is 2001106568.

7. List of published papers and reports

7.1. Published papers:

1. S.V.Zherebtsov, G.A.Salishchev, R.M. Galeyev. Dynamic recrystallization of titanium and Ti-6Al-4V two-phase titanium alloy, Proc.First Jont Int,Conf. ReX&GG, Springer-Verlag, 2001, V.2, pp.961-966.

   The features of mechanical behavior and structure formation during deformation of the titanium and the alloy Ti64 were studied. The mechanism of titanium structure formation depends on a deformation temperature. At high temperatures (>650°C) the formation of new grains occurs via migration of initial grain boundaries that is a feature of discontinuous recrystallization. During low temperature of deformation new grains are formed according to the continuous mechanism inside of initial grains via formation of subgrains with the following increase in their misorientation. The microstructure evolution in the Ti64 at studied temperatures is connected with recrystallization of phases according to the continuous mechanism and accompanied by phases spheroidization.

2. S.V.Zherebtsov, G.A.Salishchev, R.M. Galeyev, O.R.Valiakhmetov, M. F. X. Gigliotti, B. P. Bewlay, C. U. Hardwicke, R. S. Gilmore, The formation of uniform fine-grain microsructures in two-phase Ti-6Al-4V titanium alloy during hot deformation, Proc.First Jont Int,Conf. ReX&GG, Springer-Verlag, 2001, V.1, pp.569-574.

   Study of the influence of initial microstructure and thermo-mechanical regimes of deformation on formation of fine-grained microstructure in the alloy Ti-6Al-4V. It has been shown that continuous dynamic recrystallization takes place within a wide temperature range of ($\alpha+\beta$)-region. Transition of semi-coherent α/β interphase boundaries to non-coherent ones is controlled by the process of transformation of the lamellar structure to the globular one. Formation of fine-grained microstructure with minimum microtexture is possible by using corrected combination of regimes of ($\alpha+\beta$)-deformation.

3. Salishchev G.A., Murzinova M.A., Zherebtsov S.V., Galeyev R.M., Valiakhmetov O.R., Formation of nanocrystalline structure in two-phase titanium alloys by warm severe plastic deformation, *Ultrafine Grained Materials II*, Y.T. Zhu, T.G. Langton, R.S. Mishra, S.L. Semiatin, M.J. Saran, and T.C. Lowe, (eds), TMS, Warrendale, PA, 2002, PP. 113-122.

   Warm severe plastic deformation realized via multiple forging can be used for formation of fine-grained microstructure with grain size of several hundred nanometers in titanium alloys. It has been established that the least grain size not only depends on the deformation temperature, but also the phase volume fraction, phase particle size and interparticle distance.

7.2. Reports on conferences and Workshops:

1. TMS 131st Annual Meeting & Exhibition, *International Symposium on Ultrafine Grained Materials II* (Seattle, Washington,. USA, February 17-21, 2002):Salishchev G.A., Murzinova M.A., Zherebtsov S.V., Galeyev R.M., Valiakhmetov O.R., Formation of nanocrystalline structure in two-phase titanium alloys by warm severe plastic deformation.

8. Information on patents (List of patents which were obtained or may be obtained as a result of the project

On the basis of previous inventions and Project activities, prepared and filed to Russian Patent Agency the application entitled “A method for processing billets and die tool for its performance (Versions)”, by Valiakhmetov O.R., Galeyev R.M., Kaibyshev O.A., and Salishchev G.A., its priority number is 2001106568.

The invention can be used for regulated changes of structure and properties of the material , including formation of submicrocrystalline structure. The method for processing materials by deformation comprises successive stages of pressing of a billet along its height in the cavity of the

device for processing. This deformation provides plastic flow of the material along one direction and this direction does not coincide with the direction of the deforming load. At least, beginning from the second stage the billet is placed in the cavity of the devise so as to restrict the plastic flow of the material along the identified direction from one of billet's faces. After each stage the billet is withdrawn from the cavity after release, at least, its three faces.

Project Manager G.A. Salishchev